American Chemical Service, Inc. NPL Site

Pilot/Treatability Testing QAPP and Field Sampling Plans

August 1996 Submittal
with January 1997 and
June 1997 Updates

REVISION OF SECTION 5.0 OF THE PREDESIGN WORKPLAN

Page 5-14 of the predesign workplan was modified in response to the comments. The revised copy of Page 5-14 is attached.

No other changes were made to the predesign workplan dated January 1997.

scale ISVE system will be established using a combination of modeling, typical radius of influence for sites with similar lithology, and professional judgment based on the pilot study results.

5.2 LTTT AND PRETREATMENT/MATERIAL HANDLING STUDY IN THE OFCA

5.2.1. Objectives

Since the ROD requires implementation of low temperature thermal to treat the waste in the Off-Site Containment Area, the data generated from the pilot/treatability study will be used to determine design parameters for full scale implementation of the technology. Specifically, pilot/treatability testing will be conducted to:

- Evaluate whether LTTT can meet the performance standards listed in Table 5-1
- Develop LTTT operating parameters needed to treat soils and buried wastes to the performance standards.

On-site LTTT is required for full-scale treatment of dewatered/excavated buried waste materials and source areas. Treatment residuals from LTTT meeting performance standards will be redeposited on site. Air emissions from the LTTT must be controlled to meet the IDEM air quality standards and the 1×10^{-5} cumulative cancer risk range as stated in Section II.F.3 (Page 15) of the SOW. Air monitoring will be conducted while the on-site pilot study activities are taking place. The specific monitoring methods, frequency, duration, and compounds to be analyzed are discussed in Section 5.2.2.4. and in Appendix C, Section C.5 of the Pilot/Treatability Testing QAPP and FSP.

Excavation and dewatering of buried waste is required prior to using LTTT at the ACS Site. Soil borings from previous site investigations indicate that the water table is between 4 and 5 feet below ground surface and that a large quantity and wide variety of contaminated debris are present at the site, particularly in the saturated zone. These materials have the potential for significant impact on the remedial design and implementation. Consequently, a pretreatment/material handling pilot study is being conducted in addition to the required LTTT treatability study to evaluate potential material handling issues. The material handling issues are associated with the debris and wet nature of the material to be screened after excavation.

C.4.0 SOIL SAMPLING

C.4.1 SAMPLING PROCEDURE

Each test pit will have one five gallon composited sample of screened, mixed soil taken and shipped to the treatability test laboratory. An identical amount will be packaged and stored on-site in a secure location as an archive sample. Samples will be taken as follows:

Obtain three grab samples with a clean shovel from eight different locations within the screened, mixed, soil tailings pile. Place the grab samples on a plastic sheet and mix thoroughly breaking clods of soil or debris as much as possible. Quarter the sample and move it to the edges of the sheet. Mix each quarter. Roll the quarters back to the middle of the sheet and mix the entire sample again. By taking several grab samples from the screened soil pile to form the composite and by mixing the grabs thoroughly, the resulting composite will be as representative of the test pit area as possible. Finally, scoop samples into two 5 gallon hazardous waste shipping containers. Fill each sample container completely to the top, leaving no head space. Seal the container to insure airtightness, label appropriately. Affix tamper-proofing tape to the lid and container. The procedure for compositing of the soils from each test pit for purposes of thermal treatability testing is described in D3.1.3 in Appendix D of the Treatability Test QAPP.

C.4.2 SAMPLE NOMENCLATURE

Sample numbers will have the following format:

AA-P-MMDDYY

Where:

AA = Sample type (SP = soil primary, SA = soil archive)

P = Pit number (1 = Pit #1, 2 = Pit #2, etc.)

MM = Month sample was taken

DD = Day Sample was taken

YY = Year Sample was taken

Example 1: Primary soil sample taken from Pit 5 on August 10, 1996 --- SP-5-081096

Example 2: Archive soil sample taken from Pit 1 on 08/11/96 --- SA-1-081196

C.4.3 SAMPLE CUSTODY

All samples taken will be logged on a Chain of Custody form by the sampler. The chain-of-custody form contains information on the date and time of sample collection, the sampler, the project name and

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QUALITY ASSURANCE PROJECT PLAN
AND
FIELD SAMPLING PLAN
FOR
PILOT/TREATABILITY TESTING

AMERICAN CHEMICAL SERVICE, INC.
NPL SITE

GRIFFITH, INDIANA

June 1997

PREPARED FOR:

ACS RD/RA EXECUTIVE COMMITTEE

PREPARED BY:

MONTGOMERY WATSON AMERICAS, INC.

QUALITY ASSURANCE PROJECT PLAN AND FIELD SAMPLING PLAN FOR PILOT/TREATABILITY TESTING

AMERICAN CHEMICAL SERVICES INC.
NPL SITE
GRIFFITH, INDIANA

JUNE 1997

Prepared by: Montgomery Watson

Montgomery Watson Project Manager	Date
Montgomery Watson QA Officer	Date
U.S. EPA Region 5 Remedial Project Manager	Date
U.S. EPA Region 5 Quality Assurance Reviewer	Date
IEA Quality Assurance Officer	Date
Quanterra Quality Assurance Officer	Date

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
On-Site Area ISVE Pilot Study	An ISVE pilot study will be conducted in a 30 by 30 foot dewatered test cell in the On-Site Area to assess whether ISVE will remove sufficient VOC and SVOC contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Test: System Installation and Soil Sampling	The ISVE test cell will be constructed by enclosing an approximately 30x30x25 foot area with a physical barrier (either sheet pile or slurry wall). One 2-inch piezometer, PZ501, and three 1-inch piezometers, PZ502 - PZ504, will be installed in the test cell. During drilling, each piezometer boring will be continuously logged for lithologies. A pump will be installed in Piezometer PZ501 to dewater the test cell. Piezometers PZ502 - PZ504 will be used to monitor ground-water elevation during the pilot study. Purge water from Piezometer PZ501 will be stored in a tank on site and sampled prior to disposal or on-site treatment.	USCS lithologies	A	D
				Groundwater elevation (feet above NGVD)	A	D
				SW-846 8260A (VOCs) purge water	B	D
				SW-846 8270 (SVOCs) purge water	B	D
				SW-846 8081 (Pesticides/PCBs) purge water	B	D
				SW-846 8150 (Pentachlorophenol) purge water	B	D
				Metals[(a)] (SW-846 6010A) purge water	B	D
			Two SVE well borings, VEW1 and VEW2, will be drilled to approximately 11 feet and 21 feet bgs, respectively. Each boring will be continuously sampled for lithologies. One soil/waste sample from each boring will be collected during drilling to assess vertical contaminant distribution and provide pre-test baseline data for technology evaluation. One soil sample will be collected from 5 feet bgs to assess shallow soil contamination and the other will be collected from 15 feet bgs to assess deeper soil contamination. A pump will be installed in SVE well VEW2 and along with Piezometer PZ501 will be used to dewater the site. Purge water from VEW2 will be combined with effluent from PZ501.	USCS lithologies	C	TA, D
				VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	D
				Metals[(b)] (SW-846 6010A)	C	D
				Phosphorous (EPA/600 365 2)	C	D
				Hexavalent Chrome (SW-846 7196)	C	D
				TOC (SW-846 9060)	B	BA
				TKN (EPA/600 351.2)	B	BA
				o-Phosphate (EPA/600 365.2)	B	BA
				pH (field measurement)	A	BA
				Heterotrophic Plate Count	A	BA
				Soil particle size distribution (ASTM D-422)	A	D
				Moisture content	A	D
			Four soil vapor probe borings (VP1 - VP4) will be drilled to approximately 18 feet bgs. The borings will be continuously sampled for lithologies. Two soil waste samples per boring will be collected during drilling to assess vertical contaminant distribution and provide pre-test baseline data for technology evaluation. One soil sample will be collected from 5 feet bgs and the other from 15 feet bgs. Three nested soil vapor probes will be installed in each boring at depths of 7, 12, and 17 feet bgs. The letters A, B, and C will be used to identify the relative depth of each probe.	USCS lithologies	A	D
				VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Metals[(b)] (SW-846 6010A)	C	D
				Phosphorous (EPA/600 365.2)	C	D
				Hexavalent Chrome (SW-846 7196)	C	D
				TOC (SW-846 9060)	B	BA
				TKN (EPA/600 351.2)	B	BA
				o-Phosphate (EPA/600 365.2)	B	BA
				pH (field measurement)	A	BA
				Heterotrophic plate count	A	BA
				Soil particle size distribution (ASTM D-422)	A	D
				Moisture content	A	D
		Meteorological Measurements	A weather station will be set up at the test site to monitor weather conditions to assess the impact on meter calibration and soil vapor data.	Ambient air temperature	A	TA, D
				Relative humidity	A	TA, D
				Barometric pressure	A	TA, D
				Wind speed and direction	A	TA, D
				Precipitation	A	TA, D
		Pre-Test: Soil Vapor Sampling	Soil vapor samples will be collected from both extraction wells and each soil vapor probe prior to system start up to provide baseline data for biological activity evaluation.	Carbon dioxide	A	D, BA
				Oxygen	A	D, BA
				Total volatile hydrocarbons	A	D, BA
		Pre-Test: Initial Soil Vapor Sampling	Soil vapor samples will be collected from each SVE well and soil vapor probe after the first soil vapor purge volume has been extracted to provide baseline data for technology evaluation.	SVOCs (TO-13)	B	TA, D
				VOCs (TO-14)	B	TA, D
				Ethane and Methane (TO-3)	B	TA, D
		Short-Term Variable Flow Rate Test	A short term test will be conducted to determine system operation parameters. During the short term test vacuum measurements and flow rate measurements will be collected until the vacuum stabilizes.	Vacuum (inches of mercury)	A	D
				Flow rate (ft^3/minute)	A	D

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
On-Site Area ISVE Pilot Study (con't)		Long-Term Constant Flow Rate Test:	A long-term constant flow rate test will be conducted based on the results of the short-term test. During this test the system operation parameters will be monitored, soil vapor samples for TVH will be collected from the extraction well manifold and the vapor treatment off-gas system at 8-hour intervals during the first week of operation, 12-hour intervals during the second week of operation, and daily for the remainder of the pilot study. Soil vapor samples will be collected from the sampling port at the manifold and off-gas samples will be collected from the vapor treatment system for contaminant analysis every two days during the first week of operation, two times per week during the second week of operation, weekly during the third through the sixth week of operation, and monthly thereafter	Vacuum (inches of mercury)	A	D, BA
				Flow rate (ft^3/minute)	A	D, BA
				Total volatile hydrocarbons	A	D, BA
				Condensate volume	A	D
				VOCs (TO-14)	B	TA, D
				SVOCs (TO-13)	B	TA, D
				Groundwater elevation (feet above NGVD)	A	D
		Condensate Sampling	Condensate samples will be collected from the air/water separator on a bi-weekly basis for the duration of the ISVE system operation to evaluate the type and concentration of contaminants in the condensate.	SW-846 8260A (VOCs)	B	D
				SW-846 8270 (SVOC)	B	D
				SW-846 8081 (Pesticides/PCBs)	B	D
				SW-846 8150 (Pentachlorophenol)	B	D
				Metals[a] (SW-846 6010A)	B	D
		Post Test Soil Vapor Sampling	After the long-term ISVE test is complete, a 48 hour test will be conducted to assess equilibration of the test area. Soil vapor samples will be collected at each vapor probe and each SVE extraction well immediately after shut down, 1/2 hour, and at 1, 2, 4, 8, 12, 24, and 48 hours after shut down	Carbon dioxide	A	D, BA
				Oxygen	A	D, BA
				Total volatile hydrocarbons	A	D, BA
			Within 48 hours after the long-term term constant rate test, post-test soil vapor samples will be collected from each soil vapor probe and each extraction well.	SVOCs (TO-13)	B	TA, D
				VOCs (TO-14)	B	TA, D
				Ethane and Methane (TO-3)		
		Post-Test: Soil Sampling	Within two weeks of the conclusion of the constant - rate test, five soil borings, SB501 - SB505, will be drilled and sampled for technology evaluation. A boring will be located next to each soil vapor probe and one next to the SVE wells.	VOCs (SW-846 8260A)	C	TA, D
				SVOC (SW-846 8270)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Metals (SW-846 6010A)[b]	C	D
			Two soil waste samples per boring will be collected from the same depth intervals from which the pre-test soil samples were collected.	Hexavalent Chrome (SW-846 7196)	B	D
				TOC (SW-846 9060)	B	BA
				TKN (EPA/600 351.2)	B	BA
				o-Phosphate (EPA/600 365.2)	B	BA
				pH (field measurement)	A	BA
				Heterotrophic plate count	A	BA
				Soil particle size distribution (ASTM D-422)	A	D, BA
ONCA ISVE/Air Sparging Pilot Study	An ISVE/air sparging pilot study will be conducted in the ONCA to assess whether this technology will remove sufficient contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Test: System Installation and Soil Sampling	Four monitoring well (MW501 - MW504) and two air injection well (AS1 and AS2) borings will be drilled to approximately 20 feet bgs. Each boring will be continuously logged for lithologies and two soil samples per boring will be collected to assess vertical contaminant distribution and to provide baseline data for technology evaluation. One soil sample will be collected 1 foot bgs (above the water table) and the other will be collected 10 feet bgs (below the water table). In addition to the monitoring well, one soil vapor probe (VP5 - VP8) will be installed in each monitoring well boring. Horizontal SVE wells will be installed in 60 foot x18 inch x 2 foot trenches. Soil samples will not be collected during trench excavation.	USCS lithologies	A	D
				VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Metals[b] (SW-846 6010A)	C	D
				Phosphorous (EPA/600 365.2)	C	D
				Hexavalent Chrome (SW-846 7196)	C	D
				TOC (SW-846 9060)	B	BA
				TKN (EPA/600 351.2)	B	BA
				ortho-Phosphate (EPA/600 365.2)	B	BA
				pH (field measurement)	B	BA
				Heterotrophic plate count	A	BA
				Soil particle size distribution (ASTM D-422)	A	D
				Groundwater elevation (feet above NGVD)	A	D
				Moisture Content	A	D

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Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Pre-Test System Installation and Soil Sampling (con't)	Three piezometers, P505- P507 will be installed to monitor groundwater elevation near the horizontal SVE wells during the pilot test During drilling, each piezometer boring will be continuously sampled for lithologies	USCS lithologies Ground-water elevation (feet above NGVD)	A A	D D
		Meteorological Measurements	A weather station will be set up at the test site to monitor weather conditions to assess the impact on meter calibration and soil vapor data.	Ambient air temperature Relative humidity Barometric pressure Wind speed and direction Precipitation	A A A A A	TA, D TA, D TA, D TA, D TA, D
		Pre-Test Groundwater Sampling	After the monitoring wells are installed and developed groundwater samples will be collected from each well to provide baseline data for technology evaluation	VOCs (SW-846 8260A) SVOCs (SW-846 8270) Pesticides/PCBs (SW-846 8081) Pentachlorophenol (SW-846 8150) Metals (SW-846 6010A)[a] Cations[c] (SW-846 6010A) Hexavalent Chrome (SW-846 7196) TOC (415.1) Chloride (EPA/600 325.2) Fluoride (EPA/600 340.2) Sulfate (EPA/600 375.4) Sulfide (EPA/600 376.1) Nitrate/Nitrite (EPA/600 353.2) Ammonia (EPA /600 350.3) ortho-Phosphate (EPA/600 365.2) Heterotrophic Plate Count	C C C C C B C B B B B B B B B A	TA, D TA, D TA, D TA, D D BA D BA BA BA BA BA BA BA BA BA
		ISVE Pre-Test Soil Vapor Sampling	Soil vapor samples will be collected from the extraction trench manifold and soil vapor probes prior to start up of the ISVE system to provide baseline data for biological activity evaluation	Carbon dioxide Oxygen Total volatile hydrocarbons	A A A	D, BA D, BA D, BA
		ISVE Initial Vapor Sampling	Soil vapor samples will be collected from the sampling port on the extraction trench manifold and from each soil vapor probe after the first soil vapor purge volume has been extracted to provide baseline data.	SVOCs (TO-13) VOCs (TO-14) Ethane and Methane	B B B	TA, D TA, D TA, D
		ISVE Short-Term Variable Flow Rate Test	A short-term variable flow rate test will be conducted to determine system operation parameters. During the short-term test system operation parameters will be monitored.	Vacuum (inches of mercury) Flow rate (ft^3/minute)	A A	D, BA D, BA
		ISVE Two-Week Constant Rate Test	A two-week constant flow rate test will be conducted using the operation parameters from the short-term test. During this test, groundwater elevations will be measured in the piezometers and monitoring wells, soil vapor samples for system parameter assessment will be collected every 8 hours the first week and every 12 hours the second week, condensate samples for contaminant concentration assessment will be collected on a weekly basis, soil vapor samples for VOCs and SVOCs analysis will be collected from the extraction trench manifold every two days the first week of the test and twice the second week of the test, and off-gas samples for emissions evaluation will be collected from the vapor treatment unit on a bi-weekly basis.	Vacuum (inches of mercury) Flow rate (ft^3/meter) Groundwater elevation (feet above NGVD) Total volatile hydrocarbons Condensate volume VOCs (SW-846 8260A) SVOC (SW-846 8270) Pesticides/PCBs (SW-846 8081) Pentachlorophenol (SW-846 8150) Metals (SW-846 6010A)[a] SVOCs (TO-13) VOCs (TO-14)	A A A A A C C C C C B B	D D D D, BA D D D D D D D, VE D, VE
		Air Sparging Short-Term Variable Flow Rate Test	A short-term variable flow rate test will be conducted to determine system operation parameters. This test will be conducted until the system stabilizes at each flow step. Flow rates will be measured at Air Sparge Wells AS1 and AS2 and the blower.	Pressure (inches of mercury) Flow rate (ft^3/minute)	B B	D D

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Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Air Sparging Two-Week Constant Rate Test	A two-week constant flow rate test will be conducted using the operation parameters from the short-term test. During this test, groundwater elevations will be measured in the piezometers and monitoring wells and system operation parameters will be monitored	Vacuum (inches of mercury) Flow rate (ft^3/meter) Groundwater elevation (feet above NGVD) Total volatile hydrocarbons	A A A A	D D D D, BA
		ISVE/Air Sparging Two Week Constant Rate Test	The two week constant-rate test will be based on the operation parameters from the short-term tests. During this test, groundwater elevation will be monitored; samples from the extraction trench manifold and off-gas from the vapor treatment system will be collected for operating parameters every 8 hours the first week of the test and every 12 hours during the second week of the test, and soil vapor samples from the extraction trench manifold and off-gas from the vapor treatment system will be collected for VOC and SVOC analysis every two days the first week of the test and twice during the second week of the test.	Vacuum (inches of mercury) Flow rate (ft^3/minute) Groundwater elevation (feet above NGVD) Total volatile hydrocarbons SVOCs (TO-13) VOCs (TO-14)	A A A A B B	D D D D, BA D, VE D, VE
		ISVE/Air Sparging Condensate Sampling	Condensate samples will be collected from the air/water separator on a weekly basis for the duration of the ISVE/air sparging system operation.	SW-846 8260A (VOCs) SW-846 8270 (SVOC) SW-846 8081 (Pesticides/PCBs) SW-846 8150 (Pentachlorophenol) Metals (SW-846 6010A)[a]	B B B B B	D D D D D
		Post-Test: Soil Vapor Sampling	After the 2-week constant rate ISVE/air sparging test, a 48 hour test will be conducted to assess post-test equilibration of the study area. Soil vapor samples will be collected at each vapor probe and the extraction trench manifold for biological activity evaluation at shutdown, 1/2 hour and 1, 2, 4, 8, 12, 24, and 48 hours after shut down	Carbon dioxide Oxygen Total volatile hydrocarbons	A A A	D, BA D, BA D, BA
			Within 48 hours of the equilibration test, post-test soil vapor samples will be collected from each soil vapor probe and the extraction trench manifold for VOC, SVOC, ethane, and methane analysis.	SVOCs (TO-13) VOCs (TO-14) Ethane and Methane (TO-3)	B B B	TA, D TA, D TA, D
		Post-Test: Groundwater Sampling	Within 48 hours of the equilibration test, groundwater samples will be collected from each monitoring well for technology evaluation.	VOCs (SW-846 8260A) SVOCs (SW-846 8270) Pesticides/PCBs (SW-846 8081) Pentachlorophenol (SW-846 8150) Metals (SW-846 6010A)[a] Cations (SW-846 6010A) Hexavalent Chrome (SW-846 7196) TOC (415.1) Chloride (EPA/600 325.2) Fluoride (EPA/600 340.2) Sulfate (EPA/600 375.4) Sulfide (EPA/600 376.1) Nitrate/Nitrite (EPA/600 353.2) Ammonia (EPA /600 350.3) ortho-Phosphate (EPA/600 365.2) Heterotrophic Plate Count	C C C C C B C B B B B B B B B A	TA, D TA, D TA, D TA, D D BA D BA BA BA BA BA BA BA BA BA

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Post Test: Soil Sampling	Within two weeks after the long-term test has ended, six soil borings, SB506 - SB511, will be drilled and sampled for technology evaluation. One boring will be located next to each monitoring well and air sparging well. Two soil samples per boring will be collected from the same depth intervals from which the pre-test soil samples were collected.	VOCs (SW-846 8260A) SVOC (SW-846 8270) Pesticides/PCBs (SW-846 8081) Metals (SW-846 6010A)[b] Phosphorous (EPA/600 365.2) Hexavalent Chrome (SW-846 7196) TOC (SW-846 9060) TKN (EPA/600 351.2) ortho-Phosphate (EPA/600 365.2) pH (field measurement) Heterotrophic Plate Count Soil particle size distribution (ASTM D-422) Moisture Content	C C C C C C B B B B A A A	TA, D TA, D TA, D D D BA BA BA BA BA BA D D
OFCA Pre-Treatment Materials Handling Treatability Study	The pre-treatment materials handling treatability study will be conducted to assess the type and quantity of debris in soils representative of the ACS site, to assess the ability of conventional materials handling equipment to effectively separate debris from soils to be thermally treated, determine the magnitude of the VOC fugitive emissions during excavation activities, and to collect soil samples for thermal treatability testing	Soil Excavation	Five 20 x 20 test pits will be excavated. Material removed from each test pit will be screened for oversized reject material and soil. The oversized reject material will be weighed and the percentage of debris types will be estimated. The soil will be weighed and sampled for low temperature thermal treatment treatability testing.	Total tonnage per test pit Total tonnage of soil per grid Total tonnage of debris per grid Percent debris per grid Percent debris type per grid (i.e., metal) Screening throughput in tons/hour PCB Field Screening	A A A A A A A	TA, D TA, D TA, D TA, D TA, D TA, D TA
		Air Monitoring	During excavation and screening, ambient air samples will be collected upwind and downwind of material handling at eight-hour intervals on a daily basis Meteorological conditions will be monitored with an on-site weather station.	VOCs (TO -14) Ambient air temperatures Humidity Barometric pressure Wind speed and direction	B A A A A	TA, D Information Information Information Information
OFCA Low Temperature Thermal Treatment (LTTT) Treatability Study	An LTTT treatability study will be conducted on the soils collected from the pre-treatment materials handling treatability study to assess whether this technology will remove sufficient VOC and SVOC contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Treatment: Soil Analysis	Samples from soil collected during the pre-treatment materials handling study will be analyzed for VOCs. These samples will be collected as the soil is received by the thermal treatment laboratory to provide baseline VOC concentrations for technology evaluation and air emission assessment.	VOCs (SW-846 8260A)	C	TA, D
			After the VOC samples are collected, the soil will be prepared for treatment and soil samples will be collected to provide data for composite sample preparation.	VOCs (SW-846 8260A) SVOCs (SW-846 8270) Pesticides/PCBs (SW-846 8081)	C C C	TA, D TA, D TA, D
			Based on the results of the prepared sample analyses, the soil will be batched to form two composite feed samples that represent worst-case and typical conditions. Soil from the composite feed will be analyzed to provide pre-treatment baseline data for technology evaluation.	VOCs (SW-846 8260A) SVOCs (SW-846 8270) Pesticides/PCBs (SW-846 8081) Dioxins/Furans (SW-846 8290) Metals (SW-846 6010A)[d] Phosphorous (EPA/600 365.2) TCLP Metals (SW-846 1311/6010A/7000) Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142) Leachable chlorides (GLI G-40/ME-4) Ash Fusability (ASTM D1857) Soil pH (SW-846 9045) Heating value (ASTMD-1989) Particle size distribution (ASTM D-422) Atterberg limits (ASTM D-4318) Proctor density (ASTM D-698) Soil classification (ASTM D-2487)	C C C B C C B A A A A A A A A A	TA, D TA, D TA, D TA, D TA, D TA, D TA, D TA, D TA, D Information Information Information Information Information Information Information
		Tray Test	Aliquots of the composite soil samples will be thermally treated at temperatures from 700°F to 1,100°F. Post-treatment soil samples will be analyzed to determine operation criteria for the Rotary Thermal Apparatus Testing.	VOCs (SW-846 8260A) SVOCs (SW-846 8270) Pesticides/PCBs (SW-846 8081) Dioxins/Furans (SW-846 8290)	C C C B	TA, D TA, D TA, D TA, D

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Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
OFCA Low Temperature Thermal Treatment (LTTT) Treatability Study (con't)		Rotary Thermal Apparatus Testing	The composite soil samples will be thermally treated based upon the results of the Tray Tests. Post-treatment soil samples will be analyzed to provide data for technology evaluation	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Dioxins/Furans (SW-846 8290)	B	TA, D
				Metals (SW-846 6010A)[(d)]	C	TA, D
				Phosphorous (EPA/600 365.2)	C	TA, D
				TCLP Metals (SW-846 1311/6010A/7000)	B	TA, D
				Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142)	A	TA, D
				Leachable chlorides (GLI G-40/ME-4)	A	TA, D
				Soil pH (SW-846 9045)	A	Information
			Off-gas purge samples will also be analyzed during the rotary thermal apparatus testing to provide data for technology evaluation. These samples consist of off-gas solubilized in water and in methanol and sorbed to XAD™ resin and to Tenex™ resin.	VOCs (SW-846 8260A)	B	Information
				SVOCs (SW-846 8270)	B	Information
				Pesticides/PCBs (SW-846 8081)	B	Information
				Dioxins/Furans (SW-846 8290)	B	Information
				Acidity (EPA/600 310 1)	A	Information
				Water pH (EPA 150.1, SW-846 9040/9045)	A	Information
				Total sulfur (ASTM D4239-85)	A	Information
				Total chloride (EPA/600325.2/325.3)	A	Information

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).
EPA Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).
Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, April 1994
American Society for Testing and Materials, 1985.

(a) Arsenic, beryllium, thallium, manganese
(b) Antimony, cadmium, barium, lead, phosphorous, potassium
(c) Calcium, iron, magnesium, phosphorous, potassium, sodium
(d) Beryllium, thallium, sodium, potassium

ACS American Chemical Service
BA Biodegradation assessment
bgs Below ground surface
ISVE In-situ soil vapor extraction
LTTT Low temperature thermal treatment
NGVD National geodetic vertical datum
OFCA Off-Site Containment Area
ONCA On-site Containment Area
PCB Polychlorinated biphenyl
ROD Record of Decision
SOW Statement of Work
SVE Soil vapor extraction
SVOC Semi-volatile organic compound
TA Technology assessment
TCLP Toxicity characteristic leaching procedure
TKN Total Kjeldahl nitrogen
TOC Total organic compound
UAO Unilateral Administrative Order
USCS Unified soil classification system
VE Air emission assessment
VOC Volatile organic compound

RCRA metals: Arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver

A Field data, microbial data, and soil physical and chemical characteristic data
B SW-846 or EPA/600 methodologies and associated QC with standard data packages
C SW-846 methodologies and associated QC with CLP-like data packages
D Design

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
A - Field Data	• Soil data collected on site using portable meters	• pH	• Instrument calibration information • pH Data (pH units)	• Field form • Field form
	• Soil vapor data collected on site using portable meters	• Carbon dioxide (CO_2)	• Instrument calibration information • CO_2 Data (percent)	• Field form • Field form
		• Oxygen (O_2)	• Instrument calibration information • O_2 Data (percent)	• Field form • Field form
		• Total volatile hydrocarbons (TVH)	• Instrument calibration information • TPH Data (meter units)	• Field form • Field form
	• Groundwater parameters collected on site using portable meters	• Temperature	• Instrument calibration information • Temperature Data (degrees Centigrade [°C])	• Field form • Field form
		• pH	• Instrument calibration information • pH Data (pH units)	• Field form • Field form
		• Dissolved oxygen (DO)	• Instrument calibration information • DO Data (milligrams/liter [mg/l])	• Field form • Field form
		• Reduction-oxidation potential (Eh)	• Instrument calibration information • Eh Data (millivolts)	• Field form • Field form
		• Specific conductivity (SC)	• Instrument calibration information • SC Data (μmhos)	• Field form • Field form
		• Turbidity	• Instrument calibration information • Turbidity Data (Neptholometric turbidity units [NTU])	• Field form • Field form
	• Field data collected during the OFCA Pre-Treatment Materials Handling Treatability Study	• Mass measurements of soil and debris	• Total tonnage per test pit • Total tonnage of soil per grid • Total tonnage of debris per grid • Percent debris per grid • Percent debris type per grid (i.e., metal) • PCB Field screening data • Screening throughput in tons/hour	• Field form • Field form • Field form • Field form • Field form • Field form • Field form
A - Microbial Data	• Microbial population counts for soil and groundwater	• Heterotrophic plate count	• Colony forming units (CFUs)	• Data report
A - Meterological Data	• Field data collected using a field weather station	• Meterological data	• Ambient air temperature (°F) • Ambient relative humidity • Ambient barometric pressure • Ambient wind speed and direction • Precipitation	• Instrument report • Instrument report • Instrument report • Instrument report • Field form
A - Soil chemical and physical characteristic data	• ASTM methods used to assess the chemical and physical characteristics of soil during the OFCA LTTT treatability study	• Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142) • Leachable chlorides (GLI G-40/ME-4) • Total Sulfur (ASTM D-4239-85) • Ash Fusability (ASTM D1857) • Soil pH (SW-846 9045) • Heating value (ASTMD-1989) • Particle size distribution (ASTM D-422) • Atterberg limits (ASTM D-4318) • Proctor density (ASTM D-698) • Soil classification (ASTM D-2487)	• Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results	• Data report • Data report • Data report • Data report • Data report • Data report • Data report • Data report • Data report • Data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
A - Standard Methods of Analysis	EPA/600 methods and associated QC	• Acidity (EPA/600 310.1) • Water pH (EPA 150.1, SW-846 9040/9045) • Total sulfur (EPA/600 375.2/375.4/300) • Total chloride (EPA/600325.2/325.3)	• Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results	• Data report • Data report • Data report • Data report
B- Standard Methods of Analysis	• OFCA LTTT off-gas data from EPA SW-846	• VOCs (SW-846 8260A) • SVOCs (SW-846 8270) • Pesticides/PCBs (SW-846 8081) • Dioxins/Furans (SW-846 8290)	• Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results	• Data report • Data report • Data report • Data report
B - Standard Methods of Analysis	• EPA SW-846 methods and associated QC • Standard air methods and associated QC • EPA/600 methods and associated QC	• SW-846 8260A (VOCs) • SW-846 8270 (SVOCs) • SW-846 8081 (Pesticides/PCBs)	• Case narrative • Completed chain of custodies (form and internal tracking documents)	• Hard copy of data report • Hard copy of data report
		• SW-846 8150 (Pentachlorophenol) • SW-846 8290 (Dioxin/furan)	• Initial calibration summary form	• Hard copy of data report
		• SW-846 6010A (As, Ca, Be, Fe, Mg, Mn, P, K,Na, Tl)	• Continuing calibration summary form	• Hard copy of data report
		• TO-13 SVOCs (Air)	• Injection logs	• Hard copy of data report
		• TO-14 VOCs (Air) • EPA/600 325.2 (Chloride) • EPA/600 340.2 (Fluoride) • EPA/600 375.4) (Sulfate) • EPA/600 376.1 (Sulfide) • EPA/600 353.2 (Nitrate and Nitrite)	• Target compound results for all samples, including field QC samples including dilution factors, reanalysis, batching information, and bracketing information	• Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data
		• EPA/600 350.3 (Ammonia) • EPA/600 351.2 (Total Kjeldahl nitrogen)	• Method blank results	• Hard copy of data report • Electronic copy of data
		• EPA/600 365.2 (ortho-Phosphate) • SW-846 9060 (Total organic carbon, soil)	• Ambient Air Blank (air samples only)	• Hard copy of data report • Electronic copy of data
		• EPA/600 415.1 (Total organic carbon, water) • TO-3 Methane/ethane (air)	• MS/MSD results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
		• SW-846 8290 Dioxins/Furans	• LCS results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
			• Surrogate results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
			• Holding time summary	• Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
B - Standard Methods of Analysis	• SW-846 Methodologies for 40 CFR 1311 TCLP RCRA Metals	• SW-846 6010A (Ag, As, Ba, Cd, Cr, Pb, Se)	• Case narrative	• Hard copy of data report
		• SW-846 7471 (Mercury)	• Target compound results for all samples, including field QC samples	• Hard copy of data report • Electronic copy of data
			• Date sample collected, extracted, and analyzed	• Hard copy of data report • Electronic copy of data
			• Method blank results	• Hard copy of data report • Electronic copy of data
			• MS results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
			• LCS results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
C - Standard Methods of Analysis	• GC/MS SW-846 methods and associated QC with CLP-like data reports	• SW-846 8260A (VOCs) • SW-846 8270 (SVOCs)	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• QC Summary	
			—System monitoring compound summary	• Hard copy of data report
			—Surrogate results	• Hard copy of data report • Electronic copy of data
			—LCS results	• Hard copy of data report • Electronic copy of data
			—Method blank results	• Hard copy of data report
			—MS/MSD results	• Hard copy of data report • Electronic copy of data
			—GC/MS instrument Performance Check	• Hard copy of data report
			—Internal standard area and RT summary	• Hard copy of data report
			• Sample Data	
			—Target compound results	• Hard copy of data report • Electronic copy of data
			—TICs	• Hard copy of data report • Electronic copy of data
			—Reconstructed total ion chromatograms (RIC)	• Hard copy of data report
			—Raw spectra and background-subtracted mass spectra of target compounds identified	• Hard copy of data report
			• Standards Data (All instruments)	
			—Initial calibration data	• Hard copy of data report
			—RICs and quantitation reports for all standards	• Hard copy of data report
			—Continuing calibration data	• Hard copy of data report
			• Raw QC Data	
			—BFB or DFTPP/tubing standards (as appropriate)	• Hard copy of data report
			—System blank data	• Hard copy of data report
			—Laboratory control sample data	• Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
C - Standard Methods of Analysis	• GC SW-846 methods and associated QC with CLP-like data reports	• SW-846 8081 (Pesticides/PCBs) • SW-846 8150 (Pentachlorophenol, groundwater only)	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• QC Summary	
			—Surrogate results	• Hard copy of data report • Electronic copy of data
			—LCS results	• Hard copy of data report • Electronic copy of data
			—Method blank results	• Hard copy of data report • Electronic copy of data
			—MS/MSD results	• Hard copy of data report • Electronic copy of data
			• Sample Data	
			—Target compound results	• Hard copy of data report
			—Chromatograms (primary column)	• Hard copy of data report
			—Chromatograms (second column confirmation)	• Hard copy of data report
			—GC integration report of data system printout	• Hard copy of data report
			—Manual work sheets	• Hard copy of data report
			• Standards Data	
			—Initial calibration of single component analysis	• Hard copy of data report
			—Initial calibration of muticomponent analysis	• Hard copy of data report
			—Analyte resolution summary	• Hard copy of data report
			—Performance evaluation mixture	• Hard copy of data report
			—Individual standard mixture A	• Hard copy of data report
			—Individual standard mixture B	• Hard copy of data report
			—Calibration verification summary (LCP 1)	• Hard copy of data report
			—Calibration verification summary (LCP-2)	• Hard copy of data report
			—Analytical sequence	• Hard copy of data report
			—Florisil cartridge check	• Hard copy of data report
			—Pesticide identification summary for multicomponent analyte	• Hard copy of data report
			—Chromatograms and data system printouts (retention times and corresponding peak areas or peak heights	• Hard copy of data report
			• Raw QC Data	
			—System blank data	• Hard copy of data report
			—Laboratory control sample data	• Hard copy of data report
			—Florisil data	• Hard copy of data report
			—LCS data	• Hard copy of data report
			—Analytical sequence	• Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
C - Standard Methods of Analysis	• Inorganic SW-846 and EPA/600 methods and associated QC with CLP-like data reports	• SW-846 7196 (Hexavalent chromium) • SW-846 6010A - Soil (Cd, Ba, Pb, Sb) • SW-846 6010A - Water (As, Tl) • EPA/600 365.2 Phosphorous	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• Sample Data	
			—Target compound results for both native and QC samples	• Hard copy of data report • Electronic copy of data
			• Quality Control Data	
			—Initial and continuing calibration verification	• Hard copy of data report
			—CRDL standard for AA and ICP	• Hard copy of data report
			—Blanks	• Hard copy of data report
			—ICP interference check sample	• Hard copy of data report
			—Spike sample recovery	• Hard copy of data report
			—Post digest spike sample recovery	• Hard copy of data report
			—Duplicates	• Hard copy of data report
			—Laboratory control sample	• Hard copy of data report
			—Standard addition results	• Hard copy of data report
			—ICP serial dilutions	• Hard copy of data report
			—Preparation log	• Hard copy of data report
			—Analysis run log	• Hard copy of data report
			• Quarterly Verification of Instrument Parameters	
			—Instrument detection limits	• Hard copy of data report
			—ICP inter-element correction factors (Form XI, Part 1)	• Hard copy of data report
			—ICP inter-element correction factors (Form XI, Part 2)	• Hard copy of data report
			• Raw Data	
			— All raw date used to obtain each reported value including real time instrument readouts and digestion or distillation logs	• Hard copy of data report

CRDL Contract-required detection limit

(a) For this project 10 TICs will be reported for SW-846 VOCs and 20 for SW-846 SVOCs (10 for base neutrals and 10 for acids).

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
SW-846 8260A (VOCs), SW-8270 (SVOCs), SW-846 8081 (Pesticides/PCBs), SW-846 8150 (Pentachlororophenol), SW-8290 (Dioxins/Furans)							
Field QC Samples	B/C	Condensate Groundwater Soil LTTT Off-Gas	Trip Blank	Identify target analytes that may have been introduced into samples during sample handling, shipping, or storage at the laboratory	One per each cooler containing samples for VOC analysis	Two 40 milliliter (ml) amber glass vials with Teflon septum caps containing reagent-grade water and preserved to a pH of <2. Prepared by the laboratory.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
		Condensate Groundwater Soil LTTT Off-Gas	Field Duplicate	Assess sampling and analytical precision	One per 10 condensate or groundwater samples and one per 20 soil samples for each analytical method	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate organic sample analysis will be ± 50 percent.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch (20 samples) for each analytical method	Reagent-grade water that is carried through the same analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Surrogate Spikes	Assess analytical accuracy	Each sample for VOC analysis including both investigative and QC samples for each method, except for SW-846 8290	Each sample will be spiked in the laboratory with surrogate spikes in accordance with the laboratories SOPs for the respective methods.	Percent recovery will be calculated for each spiked analyte and compared to the QC limits for surrogate recoveries for each respective method.
			Matrix Spike/Matrix Spike Duplicate	Identify media interference during analysis	One per 20 samples for each media for each analytical method	Condensate and groundwater samples will be collected in triplicate. Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. Additional sample volume is not required for soils. The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess media interference in the event of MS/MSD analysis that fail QC criteria and to assess laboratory accuracy	One per analytical or extraction batch for each analytical method	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to the laboratory-established QC limits for each respective method.

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
TO-13 (SVOCs), TO-14 (VOCs), TO-3 (Ethane and Methane)							
Field QC Samples	B	Air Soil Vapor Treatment System Off-Gas	Field Duplicate	Assess sampling and analytical precision	One per 10 samples for each analytical method	Duplicate of a specific native sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate samples for organic analysis will be ± 50 percent.
		Air	Ambient Air	Identify target analytes present in the ambient air to establish background concentrations	5 percent of the total number of samples for each analysis	One Tedlar™ bag or Summa canister containing ambient air.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
Laboratory QC Samples	B		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analytical method	Hydrocarbon-free air that is carried through the same analytical process as native samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Laboratory Control Sample and Laboratory Control Sample Duplicate	To assess laboratory precision and accuracy	One per 20 samples for each media for each analytical batch	LCS are prepared by the laboratory and consist of hydrocarbon-free air spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to the suggested QC limits for the respective methods.
SW-846 6010A (ICP Metals)							
Field QC Samples	B/C	Soil Groundwater Condensate	Field Duplicate	Assess sampling and analytical precision	One per 10 groundwater samples and one per 20 soil samples for each analytical method.	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate inorganic analysis will be ± 25 percent.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analysis.	Reagent-grade water that is carried through the same sample prep and analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike/Matrix Spike Duplicate	Identify media interference during analysis	One per 20 samples for each media for each analytical method.	Condensate or groundwater samples will be collected in triplicate. Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. Additional sample volume is not required for soils. The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess media interference in the event of MS/MSD analysis that fail QC criteria and to assess laboratory accuracy	One per 20 samples for each media for each analytical or extraction batch.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to laboratory-established QC limits for each respective method.

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
TCLP Metals	**SW-846 1311/6010A/7000**						
Field QC Samples	B/C	Soil	Field Duplicate	Assess sampling and analytical precision	One per one per 20 soil samples for each analytical method.	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for organic analysis will be ± 25 percent.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analysis.	Reagent-grade water that is carried through the same sample prep and analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike	Identify media interference during analysis	One per 20 samples for each analytical method.	The leachate will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess laboratory accuracy	One per analytical or extraction batch for each analytical method.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to laboratory-established QC limits for each respective method.
Wet Chemistry SW-846 7196 (Hexavalent Chromium), SW-846 9060 (TOC Soils) EPA/600 (TOC Water, Chloride, Fluoride, Sulfate, Sulfide, Nitrate/Nitrite, Ammonia, Total Kjeldahl Nitrogen, Total Phosphorous, and ortho-Phosphate)							
Field QC Samples	B/C	Groundwater Soil	Field Duplicate	Assess sampling and analytical precision	One per 10 condensate or groundwater samples and one per 20 soil samples for each analytical method.	Duplicate of a specific native sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPDs will be compared to laboratory established RPDs for matrix duplicate analysis.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
Field QC Samples (con't)			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch (20 samples) for each analytical method.	Reagent-grade water that is carried through the same analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike	Identify media interference during analysis	One per 20 samples for each media for each analytical method.	Condensate and groundwater samples will be collected in triplicate Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. Additional sample volume is not required for soils The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
		Groundwater Soil	Matrix Duplicate	Assess precision of the analytical method	One per analytical or extraction batch for each analytical method.	Groundwater samples will be collected in duplicate. One of the samples will be used for matrix duplicate analysis. Additional sample volume is not required for soils. The samples will be analyzed in accordance with laboratory SOPs.	RPD will be calculated and compared to the laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess analytical accuracy	One per analytical or extraction batch for each analytical method, except EPA-SW 7196.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with target analytes as specified in the laboratory SOPs.	Percent recovery for each spiked analyte will be calculated and compared to QC limits based on historical laboratory performance data.

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).

EPA Methods Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE Pre-Test Soil Samples																				
ISVE-VEW1	ISVE-VEW1-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-VEW2	ISVE-VEW2-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-VP1	ISVE-VP1-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-VP1-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-VP2	ISVE-VP2-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-VP2-SO-5 MS/MSD			SO	MS/MSD	NS	NS		1	1	1	NS	1	1	1	NS	NS	NS	NS	1
	ISVE-VP2-SO-5 MS/MD			SO	MS/MD	NS	NS		NS	NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	ISVE-VP2-SO-5 MD				MD				NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
	ISVE-VP2-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-VP3	ISVE-VP3-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-VP3-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-VP550-SO-5			SO	FD	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-VP4	ISVE-VP4-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-VP4-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
Total Pre-ISVE Soil Samples																				
	Investigative	NA	NA	SO	INV	NS	NS		10	10	10	NS	10	10	10	NS	10	10	10	10
	Field Duplicates	NA	NA	SO	FD	NS	NS		1	1	1	NS	1	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS		1	1	1	NS	1	1	1	NS	NS	NS	NS	1
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS		NS	NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	Matrix Duplicate	NA	NA	SO	MD	NS	NS		NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE—Initial Vapor Sampling																				
ISVE-VEW1	ISVE-VEW1-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VEW2	ISVE-VEW2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1A	ISVE-VP1A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1B	ISVE-VP1B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP51B-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1C	ISVE-VP1C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2A	ISVE-VP2A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP52A-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2B	ISVE-VP2B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2C	ISVE-VP2C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3A	ISVE-VP3A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3B	ISVE-VP3B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3C	ISVE-VP3C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4A	ISVE-VP4A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4B	ISVE-VP4B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4C	ISVE-VP4C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Pre-ISVE Soil Vapor Samples																				
	Investigative	NA	NA	SV	INV	14	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	2	2	2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Soil Vapor Samples																				
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-EV50-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Soil Vapor Samples																				
	Investigative	NA	NA	SV	INV	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Soil Vapor Treatment Off-Gas Samples																				
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-TV50-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[b]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Soil Vapor Treatment Off-Gas Samples																				
	Investigative	NA	NA	SV	INV	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Condensate Samples																				
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD50-WG			WG	FD	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[b]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Condensate Samples																				
	Investigative	NA	NA	WG	INV	NS	NS	NS	13	13	13	13	13	13	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	1	1	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE Post Test Soil Vapor Samples																				
ISVE-VEW1	ISVE-VEW1-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VEW2	ISVE-VEW2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VEW51-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1A	ISVE-VP1A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1B	ISVE-VP1B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1C	ISVE-VP1C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2A	ISVE-VP2A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2B	ISVE-VP2B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2C	ISVE-VP2C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3A	ISVE-VP3A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3B	ISVE-VP3B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3C	ISVE-VP3C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP54C-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4A	ISVE-VP4A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4B	ISVE-VP4B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4C	ISVE-VP4C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE Post Test Soil Vapor Samples																				
	Investigative	NA	NA	SV	INV	14	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total ISVE Post Test Soil Samples																				
ISVE-SB500	ISVE-SB501-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-SB501-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-SB501	ISVE-SB502-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-SB502-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-SB552-SO-15				FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-SB502	ISVE-SB503-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-SB503-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-SB503	ISVE-SB504-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	ISVE-SB504-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-SB504	ISVE-SB505-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
ISVE-SB504	ISVE-SB505-SO-6 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	NS	NS	1
	ISVE-SB505-SO-7 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	ISVE-SB505-SO-7 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
ISVE-SB504	ISVE-SB505-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
Total Post-ISVE Soil Samples																				
	Investigative	NA	NA	SO	INV	NS	NS	NS	10	10	10	NS	10	10	10	NS	10	10	10	10
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	1	1	1	NS	1	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
Purge Water Sampling																				
ISVE-WP	ISVE-WP			WG	WG	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS
Trip Blanks[c]																				
	ISVE-TB-(date)-(number)[d]			WQ	TB	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Ambient Air Samples[e]																				
	ISVE-AA1-(date)-1			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-2			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-3			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-4			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

(a) Sampled every 2 days during week 1 (3 samples), twice during week 2 (2 samples), weekly from week 3 — 6 (4 samples), and monthly for the rest of the test (5 samples assuming a 6-month test).

(b) Samples collected biweekly for the duration of the test (13 samples total assuming a 6-month test).

(c) The number of trip blanks will be dependent on the duration of the field program. One trip blank will be shipped in each cooler that contains soil or water samples for VOC analysis.

(d) Trip blanks will be designated "TB" followed by the date, and by a sequential number for trip blanks collected on a given day (i.e., the first trip blank on any day will be 1, the second 2, etc.).

(e) One ambient air sample will be collected at the beginning of the study, and then will be collected at a frequency of five percent of the total number of soil vapor samples collected.

Note: Additional sample volume is not required for MS/MSD and MS/MD analyses for soil samples.

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium-Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Pre-ISVE/Air Sparging Soil Samples																									
VEAS-MW501	VEAS-MW501-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW501-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW502	VEAS-MW502-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW502-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW503	VEAS-MW503-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW503-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW504	VEAS-MW504-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW550-SO-1			SO	FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW504-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-AS1	VEAS-AS1-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AS1-SO-10 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-AS2	VEAS-AS2-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS2-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
Total Pre-ISVE/Air Sparging Soil Samples																									
	Investigative	NA	NA	SO	INV	NS	NS	NS	12	12	12	NS	12	12	12	NS	NS	12	NS	NS	12	NS	NS	12	12
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Pre-ISVE/Air Sparging Groundwater Samples																									
VEAS-MW501	VEAS-MW501-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW551-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW502	VEAS-MW502-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW502-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-MW502-WGMS/MD			WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
VEAS-MW503	VEAS-MW503-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW504	VEAS-MW504-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Pre-ISVE/Air Sparging Groundwater Samples																									
	Investigative	NA	NA	WG	INV	NS	NS	NS	4	4	4	4	NS	NS	NS	NS	4	NS	4	4	NS	4	4	4	4
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE System Startup—Soil Vapor Samples (Air sparging system not in operation)																									
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP5	VEAS-VP5-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP6	VEAS-VP6-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP7	VEAS-VP7-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-VP54-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP8	VEAS-VP8-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE System Startup Soil Vapor Sample																									
	Investigative	NA	NA	SV	INV	5	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Soil Vapor Samples																									
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-EV51-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Soil Vapor Samples																									
	Investigative	NA	NA	SV	INV	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Vapor Treatment System Off-Gas Samples																									
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-TV250-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Vapor Treatment System Off-Gas Samples																									
	Investigative	NA	NA	SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Condensate Samples																									
VEAS-CD2	VEAS-CD2-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD250-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-CD2	VEAS-CD2-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Condensate Samples																									
	Investigative	NA	NA	WG	INV	NS	NS	NS	2	2	2	2	NS	NS	NS	2	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Air Sparging System Start-up (ISVE not in operation)—No Samples Required																									

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Combined ISVE/Air Sparging Two-Week Test—Soil Vapor Samples																									
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-EV2-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Soil Vapor Samples																									
	Investigative	NA	NA	SV	INV	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Combined ISVE/Air Sparging Two-Week Test—Vapor Treatment System Off-Gas Samples																									
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-TV51-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Vapor Treatment Off-Gas Samples																									
	Investigative	NA	NA	SV	INV	2	2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Combined ISVE/Air Sparging Two-Week Test—Condensate Samples																									
VEAS-CD2	VEAS-CD2-WG			WG	1	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2500-WG			WG	FD	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-CD2	VEAS-CD2-WG			WG	1	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium-Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Condensate Samples																									
	Investigative	NA	NA	WG	INV	NS	NS	NS	2	2	2	NS	NS	NS	NS	2	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Soil Vapor Samples																									
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP5	VEAS-VP5-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-VP63-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP6	VEAS-VP6-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP7	VEAS-VP7-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP8	VEAS-VP8-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Post-Test Soil Vapor Sampling																									
	Investigative	NA	NA	SV	INV	5	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Groundwater Sampling																									
VEAS-MW501	VEAS-MW501-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW502	VEAS-MW502-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW552-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW503	VEAS-MW503-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW504	VEAS-MW504-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW504-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-MW504-WG MS/MD			WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Post-Test Groundwater Samples																									
	Investigative	NA	NA	WG	INV	NS	NS	NS	4	4	4	4	NS	NS	NS	NS	4	NS	4	4	NS	4	4	4	4
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Soil Sampling																									
VEAS-SB506	VEAS-SB506-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB506-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB507	VEAS-SB507-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB507-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB508	VEAS-SB508-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB508-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB509	VEAS-SB509-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB509-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB551-SO-10				FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB510	VEAS-SB510-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB510-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB511	VEAS-SB511-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB511-SO-1 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-SB511-SO-1 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB511-SO-1 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-SB511-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
Total Post-Test Soil Samples																									
	Investigative	NA	NA	SO	INV	NS	NS	NS	12	12	12	NS	12	12	12	NS	NS	12	NS	NS	12	NS	NS	12	12
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Trip Blanks[a]																									
	VEAS-TB-(date)-(number)[b]			WQ	TB	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO-14)	SVOCs-Air (TO-13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8150)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, K	Phosphorous-Soil EPA/600 365.2	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Ambient Air Samples[c]																									
	VEAS-AA2-(date)-1			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-2			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-3			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-4			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-5			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-6			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-7			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Purge Water from Test Area																									
VEAS-WP	VEAS-WP			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS

NA Not applicable
NS Not scheduled

Refer to Tables A-2 and B-2 for a description of the sample identification number.

(a) The number of trip blanks will be dependent on the duration of the field program. One trip blank will be shipped in each cooler that contains soil or groundwater samples for VOC analysis.
(b) Trip blanks will be designated "TB" followed by the date the blank was collected, and by a sequential number for trip blanks collected on a given day (i.e., the first trip blank on any day will be 1, the seco
(c) One ambient air sample will be collected at the beginning of the study, and then will be collected at a frequency of five percent of the total number of soil vapor samples collected.

Note: Additional sample volume is not required for MS/MSD and MS/MD analyses for soil samples.

4.0 SAMPLING PROCEDURES

4.1 SAMPLE COLLECTION PROCEDURES

During the pilot and treatability studies soil, groundwater, condensate, soil vapor, and treatment off-gas samples will be collected. As described in the FSPs (Appendices A through D) standard procedures will be used for sample collection to meet the project DQOs. The SOPs for soil, groundwater, condensate, and soil vapor sample collection for the ISVE and ISVE/Air Sparging pilot studies are included in Appendices F and G. Sample designation criteria for these studies are described in Sections A.4.0. and B.4.0. of the Appendices A and B. The procedures for sample collection during the Pre-Treatment Materials Handling pilot study and the LTTT treatability study are described in Section 4.0 of Appendix C and Section 4.0 of Appendix D, respectively. The following sections describe sample collection order, the types of containers required for sample collection, and the procedures for QC sample collection.

4.1.1. Sample Collection Order and Containers

For all soil, groundwater, or condensate samples the sample containers will be filled in order of compound volatility or stability as follows:

1. VOCs (SW-846 8260A)
2. Hexavalent chromium (SW-846 7196)
3. SVOCs (SW-846 8270)
4. Pentachlorophenol (SW-846 8150)
5. Pesticides/PCBs (SW-846 8081)
6. Total Organic Carbon (SW-846 9060 and EPA/600 415.1)
5. Dioxins/Furans (SW-846 8290)
6. Metals and Cations (SW-846 6010A/7000)
7. Anions: chloride, fluoride, sulfate, sulfide, TKN, nitrate/nitrite, ammonia, ortho-phosphate (EPA/600)
8. Soil samples for geophysical, chemical and physical characteristics, microbial population count analysis, or groundwater samples for microbial population count analysis.

TABLE 4-1

STANDARD UNITS OF MEASUREMENT AND HOLDING TIME

Analysis (Method)[a]	Sample Container	Preservative	Unit of Measure	Holding Time
GROUNDWATER AND CONDENSATE				
VOCs (SW-846 8260A)	2 40-ml amber glass bottles with a Teflon™ septum cap; No head space	HCl; pH < 2 Chill to 4°C	µg/l	14 days from sample collection to analysis
SVOCs (SW-846 8270)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Pentachlorophenol (SW-846 8150)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Pesticides/PCBs (SW-846 8081)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Metals[b] (SW-846 6010A)	1 1-liter polyethylene bottle with a Teflon™ lined cap	HNO_3; pH < 2 Chill to 4°C	µg/l	180 days from sample collection to analysis
Ortho-Phosphate (EPA/600 365.2)	50 ml polyethylene with a Teflon™ lined cap	Filter immediately Chill to 4°C	mg/l	48 hours from sample collection to analysis
Nitrate/Nitrite (EPA/600 353.2)	100-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Ammonia (EPA/600 350.3)	500-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Chloride (EPA/600 352.2)	50-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Fluoride (EPA/600 340.2)	500-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Sulfate (EPA/600 375.4)	50-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Sulfide (EPA/600 376.1)	500-ml polyethylene bottle with a Teflon™ lined cap	HCl; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
TOC (EPA/600 415.1)	50-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Heterotrophic plate count	Sterile 125-ml polyethylene container	0.5 ml $Na_2S_2O_3$	CFU	30 hours from sample collection to analysis
SOIL				
VOCs (SW-8260A)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/kg	14 days from sample collection to analysis
SVOCs (SW-846 8270)	8-oz wide-mouth glass jar with a Teflon™ lined cap	No preservation Chill to 4°C	µg/kg	14 days from sample collection to extraction 40 days from sample extraction to analysis
Pesticides/PCBs (SW-846 8081)	Included in SVOCs container	No preservation Chill to 4°C	µg/kg	14 days from sample collection to extraction 40 days from sample extraction to analysis
Dioxins/Furans (SW-846 8290)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/kg	30 days from sample collection to extraction 40 days from sample extraction to analysis
Metals[b] (SW-846 6010A) and Phosphorous (EPA/600 365.2)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/g	180 days from sample collection to analysis
Metals[c] (SW-846 6010A) and Phosphorous (EPA/600 365.2)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/g	180 days from sample collection to analysis

TABLE 4-1

STANDARD UNITS OF MEASUREMENT AND HOLDING TIME

Analysis (Method)[a]	Sample Container	Preservative	Unit of Measure	Holding Time
SOIL (con't)				
Mercury (SW-7470)	Included in metals container	No preservation Chill to 4°C	μg/g	28 days from sample collection to analysis
RCRA metals	4-oz glass jar	No preservation Chill to 4°C	μg/l	180 days from sample collection to analysis
Hexavalent Chromium (SW-846 7196)	4-oz glass jar; No head space	No preservation Chill to 4°C	μg/g	48 hours from sample collection to extraction, 24 hours from extraction to analysis
Ortho-Phosphate (EPA/600 365.2)	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	48 hours from sample collection to analysis
Total Kjeldahl Nitrogen	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	28 days from sample collection to analysis
TOC (SW-846 9060)	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	28 days from sample collection to analysis
pH	Included in hexavalent chromium container	No preservation	pH units	Analyze immediately
Heterotrophic plate count (9215B)	4-oz sterilized glass container	Chill to 4°C	CFU	30 hours from sample collection to analysis
Soil Particle Size Distribution (ASTM D-422)	4-inch brass sleeve	None	Percent	None
AIR				
TO-13 SVOCs	PUF/XAD-2 Cartridge	No preservation Chill to 4°C	μg/sample	7 days from sample collection to analysis and 30 days from extraction to analysis
TO-14 VOCs	Tedlar™ Bag or Summa Canister	None	ppbv	3 days from sample collection to analysis (Tedlar Bag) 14 days from sample collection to analysis (Summa Canister)
TO-3 Ethane and Methane	Tedlar™ Bag or Summa Canister	None	ppbv	3 days from sample collection to analysis (Tedlar Bag) 14 days from sample collection to analysis (Summa Canister)

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).
EPA Methods Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).
Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air, April 1994.

RCRA metals: Arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver

CFU Colony-forming unit
mg/kg Milligram per kilogram
μg/l Microgram per liter
ppbv Part per billion by volume
mg/l Milligram/liter

(a) Arsenic, beryllium, thallium, manganese, calcium, iron, magnesium, phosphorous, potassium, sodium
(b) Antimony, cadmium, barium, lead, potassium
(c) Beryllium, antimony, thallium, sodium, potassium

12.2.1. Laboratory Data Validation Procedures

All Type C data will be validated and qualified in accordance with the *USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review* (Functional Guidelines) (EPA, 1994). For all data from those methodologies not included in the Functional Guidelines (Data Type B), data validation will be based on the results of QC sample analysis, sample holding time evaluation, and the basic principles for data validation outlined in the EPA guidance (i.e., method blank contamination). As described in Section 3.0, data validity will be assessed in terms of precision, accuracy, representativeness, completeness, and comparability. The calculations that will be used to assess precision, accuracy, and completeness are described in Sections 3.2.1., 3.2.2., and 3.2.5., respectively. As part of the data validation process, 10 percent of the total number of Data Type B and C data packages will be submitted to U.S. EPA Region V for an independent third party data validation. The QC data required for Type B and C data validation are listed in Table 3-1, and QC samples that will be used for data validation for each data type are listed in Table 3-2. The data qualifiers that will be used for this project are listed in Table 12-1.

As discussed previously, PARCC parameters will be used to validate the quality of analytical data and determine whether the DQOs of the project have been met. Table 3-3 depicts how the QC samples will be used to assess PARCC parameters.

REVISION OF ATTACHMENT A OF THE PILOT/ TREATABILITY TESTING QAPP AND FIELD SAMPLING PLAN

The attached SOPs have been added to Attachment A of the Pilot/Treatability Testing QAPP and Field Sampling Plan.

ATTACHMENT A

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4. QUANTERRA SOP FOR TO-3 ANALYSIS

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF TOTAL VOLATILE PETROLEUM HYDROCARBONS AND BTEX IN AIR SAMPLES (EPA METHOD TO-3)

(SUPERSEDES: NONE)

Prepared by: Val Mallari

Reviewed by: ________________________________
Chemist, Dave Olson

Approved by: ________________________________
Quality Assurance Manager,Sevda Aleckson

Approved by: ________________________________
Environmental Health and Safety Coordinator, ,Manny Velazquez

Approved by: ________________________________
Laboratory Manager, Val Mallari

evaluation process, access to these documents shall not be give to those parties unless those parties also specifically agree to these conditions.

1. SCOPE AND APPLICATION

1.1. This method is limited to the analysis of total volatile petroleum hydrocarbons and volatile aromatic hydrocarbons (benzene, toluene, ethylbenzen, xylenes or BTEX) in air samples collected in Tedlar bags or SUMMA Canisters

1.2. Also applicable to this method is the analysis of other volatile organics which elute approximately between n-pentane and n-decane and are detectable by the flame ionization and photoionization detectors, such as jet fuel (JP4) and C5 to C10 carbon chain speciation.

2. SUMMARY OF METHOD

2.1. The air samples are collected into SUMMA canisters or Tedlar bags. Tedlar bag samples can be attached to the analytical system directly without any preparation, unless a dilution is required. The SUMMA canister sample is usually received at negative or ambient pressure, which requires that it first be pressurized with nitrogen so that it can be introduced into the analytical system.

2.2. The sample is introduced into the analytical system via a sample loop. For Tedlar bag samples a vacuum pump is used to pull sample through the sample loop, while a SUMMA canister uses the pressure in the can to push sample through the loop. The loop is allowed to equilibrate for a few seconds at ambient pressure.

2.3. The loop is attached to a gas chromatograph equipped with a PID and FID in series. The FID is used to detect the hydrocarbons while the PID is used to detect the aromatic compounds.

2.4. Once the loop has equilibrated, the carrier gas of the GC is diverted through the sample loop whereby the sample is swept onto the GC column. A temperature program is used to separate the components. A computer data aquisition system accumulates the data and quantitates and reports the results.

3. DEFINITIONS

3.1. **SUMMA Canister** - SUMMA-passivation is a process by which the interior of a stainless steel, spherical container is deactivated and rendered inactive to most volatile organic compounds. This results in a highly inert sample container.

3.2. **Variable Flow Regulator (VFR)** - used during composite air sampling of SUMMA canisters. Controls the flow to the canister via a needle valve and diaphragm.

3.3. **Tedlar bag** - an inert, plastic-like bag used to collect volatile organic compounds.

4. INTERFERENCES

4.1. Due to the non-specificity of the FID detector, compounds detected may include other volatile, combustible organic compounds and may be included in the calculation of TVPH.

4.2. Many compounds in a complex hydrocarbon matrix or fuel are ionizable by the PID and therefore are detected. The resulting complexity of the chromatogram increases the likelihood of false positives for BTEX. Second column confirmation can be used, but is also susceptible to the same interferences. If unambiguous confirmation is required, GCMS can also be used to verify positive hits.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonable achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Varian Model 3400 Gas Chromatograph equipped with PID and FID in series, capable of subambient temperature ramping.

6.2. DB-624 30 meter capillary column.

6.3. PE Nelson Turbochrome GC software, version 4.0 or greater. DEC personal computer or equivalent, at least a 486/33 CPU, with 8 MB of RAM.

6.4. Stainless steel sample loops: variety of sizes 0.5ml to 100ml.

6.5. Ultra high purity Helium (99.99%), hydrogen, air, and nitrogen.

6.6. Liquid nitrogen @ ~15 psig delivery pressure.

6.7. Tedlar bags and SUMMA canisters for preparation of standards and dilution of samples.

6.8. Variable Flow Regulator capable of controlling flow from ~3ml/min to ~500ml/min.

6.9. Gastight syringes: 1 liter, 50 ml, 5 ml, 25 ml.

6.10. Pressure gauges capable of reading 30" of mercury to 30 psig

7. REAGENTS AND STANDARDS

7.1. Certified BTEX gas mixtures from Scott-Specialty gases. Nominal 50 ppmv.

7.2. Neat, unleaded gasoline.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Ambient air samples can be collected in Tedlar bags using a VOC-free pump or by placing the bag in an airtight box and then evacuating the box with a pump. The resulting vacuum in the box expands the Tedlar bag and draws sample into the bag.

8.2. An instantaneous ambient air sample can be collected in a SUMMA canister simply be opening the valve on the evacuated can and waiting a few minutes for the sample to be drawn. A composite ambient air sample is collected over a period of time using a variable flow regulator which controls the flow entering the canister. The VFR is calibrated by the laboratory for a specific duration or flow.

8.3. Air samples do not require preservatives, but should be stored in a cool, dark, secured area. Tedlar bags are placed in sturdy boxes for further protection.

8.4. Recommended holding-time for a sample collected in a SUMMA canister is 14 days, while a Tedlar bag sample has 3 days.

9. QUALITY CONTROL

9.1. Initial Calibration

9.1.1 A minimum of a five-point calibration is required prior to analysis. The relative standard deviation must be less than or equal to 25%, or the coefficient of correlation must be greater than 0.995.

9.2. Continuing Calibration Verification

9.2.1 A continuing calibration verification standard must be analyzed prior to each analytical sequence and at the end of the analytical sequence. The relative percent difference must be less than or equal to 25%.

9.3. Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

9.3.1 A laboratory control sample must be analyzed for every 5% of samples analyzed. The percent recovery of the LCS analysis must be between 80 - 120 % or established QC criteria. The QC criteria are updated once per year.

9.3.2 A laboratory control sample duplicate is analyzed for every 5% of samples analyzed. The RSD of the LCSD must be within 20% or within established QC Criteria limits. A sample duplicate can also be analyzed in lieu of an LCSD.

9.4. Method Blank

9.4.1 A method blank is analyzed after high level samples or after QC samples such as the CCV or LCS/LCSD.

9.4.2 All analytes of interest must be below the reporting limit of the method before analyses can proceed.

9.5. Out-of-Control Situations

9.5.1 Should an out-of-control situation arise, the analyst must generate an Anomaly Form to record the details of the event. The QC manager or supervisor must immediately be notified and the Anomaly Form signed by both parties. A determination is made as to the appropriate corrective action. In some cases, such as holding-time violations, the client is contacted for further action.

9.5.2 The out-of-control situation must later be followed-up to ensure that all necessary corrective-actions have been implemented. The follow-up activity is also recorded on the Anomaly Form.

10. CALIBRATION AND STANDARDIZATION

10.1. Prior to analysis, the system must be calibrated and demonstrated to be within acceptable calibration guidelines. A five-point calibration is performed where the lowest level used is near the expected method detection limit, usually ~5 times the MDL, and the highest level is determined to be the point at which linearity is lost. The concentration of the low level standard is defined to be the Reporting Limit of the method.

10.2. The acceptability of the calibration can be determined using the standard deviation of the response factors or the correlation coefficient of the linear regression of the areas vs. concentration. At each level, the response factor of each analyte is calculated. The response factors are averaged and the relative standard deviation determined (RSD). A calibration is acceptable if the RSD of the response factors is less than 25%. A calibration based on a linear regression analysis is acceptable if the coefficient of correlation is greater than 0.995.

10.3. Each day, prior to analysis, a continuing calibration verification (CCV) standard is analyzed to determine if the initial calibration is still valid. The CCV is valid for 24-hours and must be re-analyzed before more samples can be analyzed. The calibration is acceptable if the RPD is less than 25%.

11. PROCEDURE

11.1. The instrument is set-up using the parameters listed in Section 17. The appropriate QC is performed prior to analysis of samples to verify method performance.

11.2. A 5-ml sample loop is installed into the valve oven. The 5-ml loop is the default loop size and is the loop which is used to perform all calibrations; therefore, analytical results of samples run on different loop sizes must take into account the "loop" dilution factor (DF = 5ml÷sample loop volume (ml)).

11.3. Before the sample is analyzed, check to see if there is any screening data available from which to estimate a possible dilution. Screening data can come from a variety of places. Sometimes the chain-of-custody or other paperwork submitted with the samples contains field instrument readings for total hydrocarbons which can give some indication of contaminant levels. If the sample was submitted for other analyses such as total hydrocarbons as hexane, TO-14, and fixed gases, check to see if the other analyses have already been performed, and evaluate the data if available

11.4. To dilute samples, several techniques can be used. The most common technique is to transfer an aliquot of the sample into a Tedlar bag and then diluting to volume with nitrogen or air. For very large dilutions, the diluted sample can be diluted again into another Tedlar bag. Another technique is the syringe dilution. An aliquot of sample is drawn into a syringe and then the plunger of the syringe is pulled to its full volume causing the sample to be diluted. A luer-lok valve is attached to the syringe's needle port and closed to allow the contents of the syringe to equilibrate, approx. 1-2 minutes. The contents of the syringe can be transferred to another Tedlar bag for analysis, or further dilutions can be performed by depressing the syringe plunger to a known volume and then drawing in diluent gas to a final known volume. Large dilutions can be attained using this serial dilution technique.

11.5. Once the proper dilution has been performed, the sample container is attached to the sample port of the GC system and the analytical run started. The sample loop is flushed with the sample for a time which guarantees saturation of the loop with sample. Usually 3-5 times the volume of the loop is sufficient.

11.6. After a known amount of time, a valve is actuated which diverts the flow of carrier gas through the sample loop and sweeps the contents onto the GC column which is initially at subambient temperature to aid in trapping the lighter compounds.

11.7. Upon completion of the analytical run, the data system prints a report which the analyst must review for chromatography, signal saturation, retention times, interferences, and other information

11.8. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.9. Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. After the analysis has been performed, a real-time review of the data is performed to determine if a successful run has been achieved.

12.2. The chromatogram is reviewed to determine if the proper dilution has been performed. Any peaks which exceed the upper calibration of the method or which exceeds the signal output of the detector must be further diluted and re-analyzed. In the case of TVPH analysis, some peaks may experience some minor signal saturation, but not significantly affect the total peak area, and still be under the upper calibration range. The PID can experience substantial interference from TVPH compounds and make positive identification difficult even with second-column confirmation. In some cases, the reporting limit may have to be elevated to compensate for the interferences. A GCMS analysis can be used to confirm PID results.

12.3. If the sample was analyzed at the proper dilution, the chromatogram is evaluated to determine if proper integration was performed. In highly complex TVPH matrices, the baseline can be drawn incorrectly affecting the accuracy of the peak area calculation. For a TVPH as gasoline calculation to be accurate, a common baseline must be used and valley to baseline peak integration areas utilized to take advantage of total peak area of the chromatogram. The summation of the peak areas between C5 and C10 are used for all TVPH calculations.

12.4. A comparison of the TVPH chromatogram against the gasoline reference is performed to determine if the pattern is indicative of gasoline. If not, a result is still reported, but a qualifier is added to the report to reflect that the chromatographic pattern is not indicative of the presence of gasoline.

12.5. The PID chromatogram is evaluated for proper integration, also, and the retention times are evaluated for proper identification of the BTEX compounds.

13. METHOD PERFORMANCE

14. POLLUTION PREVENTION

15. WASTE MANAGEMENT

16. REFERENCES

16.1. "Measurement of Gaseous Organic Compound Emissions by Gas Chromatography," **40 CFR Part 60, Appendix A, Method 18.**

16.2. "Non-Halogenated Volatile Organics," Revision 1.0, December 1987, **Test Methods for Evaluating Solid Waste, SW-846, November 1986, Third Edition**

16.3. "The Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection," **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-89/017, June 1988**

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Operating Parameters

Oven ramp:

Initial temp. (°C)	-20
Initial time (min.)	4
Ramp 1	30°C/min.
Final temp 1	35
Temp 1 hold	1
Ramp 2	10°C/min.
Final temp 2	100
Temp 2 hold	1
Ramp 3	30°C/min.
Final temp 3	180
Temp 3 hold	3

Injector temp	150
Detector temp	220
Det A (PID)	8×10^{11}

Det B (FID) 8×10^{12}

17.2. Relays

5ml loop installed in the relay #4 position

Time	Relay	
Initial	-1-2-34	Initial setting
0.01 min.	-1-234	Turns on pump, fill sample loop
0.90 min.	-1-2-34	Stop sampling
0.95 min.	1-2-34	Inject sample, sweep loop into column
3.0 min.	-1-2-34	Stop sweeping loop, set to initial condition

17.3.

5. IEA SOP FOR LOG IN OF COMMERCIAL SAMPLES

6. IEA SOP FOR COMMERCIAL SOLVENT APPROVAL

7. IEA SOP FOR SW-846 8260A VOCs ANALYSIS

8. IEA SOP FOR SW-846 8260A VOCs LOW CONCENTRATION ANALYSIS

11. IEA SOP FOR SW-846 8270 SVOCs ANALYSIS

14. IEA SOP FOR SW-846 8081 PESTICIDES/PCBs IN SOIL AND WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director ____________________

Quality Assurance Manager ____________________ DRAFT

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in multimedia, multi-concentration samples.

2.2 Refer to Table 6 of this SOP for parameters.

2.3 The document control number for this SOP is GCSDRAFT.NC.

3.0 SUMMARY OF METHOD

3.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides and polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

- EPA Method 8081 (Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique)
- EPA Method 8000A (Gas Chromatography)

4.0 INTERFERENCES

4.1 Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

4.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract using TBA-Sulfite. Refer to the SOP for Sulfur Removal, IEA Doc# GCS01500.NC.

4.3 If an extract is to be analyzed for PCB's only, then an optional sulfuric acid cleanup may be performed. This cleanup procedure will remove contamination which may interfere with the analysis of the eluting PCB's, however, the procedure will also cause degradation of other pesticide and surrogate compounds. If contamination, due to sample matrix, is suspected in the sample, or the original extract analysis exhibits interferences, then the extract may be cleaned up with concentrated sulfuric acid and reanalyzed. A portion of the original method blank is also acid cleaned and analyzed.

4.3.1 Acid cleanup is performed by adding one part sulfuric acid to two parts of the hexane extract in a clean vial. Tighten the vial and agitate the sample for thirty seconds. After cleanup, pipet the extract layer to another vial. Repeat the cleanup procedure until the acid layer is light brown to orange in color. Complete the acid cleanup procedure by performing a sulfur cleanup. This cleanup must also be performed on the corresponding method blank and QC if all samples required cleanup. Refer to the SOP for Sulfur Removal, IEA Doc# GCS01500.NC. Document in the case narrative each sample on which acid and/or sulfur cleanup was performed.

4.3.2 Florisil cleanup is an optional clean procedure used as necessary to eliminate interfering compounds. Refer to USEPA CLP OLMO3.1 Statement of Work, pg. D-53/Pest, Section 10.1.8.2 on Florisil cleanup using Florisil Cartridges.

5.0 SAFETY

5.1 All compounds listed in Tables 1-7 (see attached documents) along with their respective solvents (i.e., hexane, isooctane, acetone, toluene) are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

5.2 Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Soil samples are collected with 250 ml or 500 ml amber glass containers with Teflon-coated liner.

6.1.2 Water sample are collected in 2x1 liter amber glass containers with Teflon-lined lids.

6.1.3 Precleaned certified sample bottles are purchased from a commercial vendor.

6.1.4 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

6.4.1 Soil samples must be extracted within 14 days from sampling.

6.4.2 Water samples must be extracted within 7 days from collection.

6.4.3 Extracts must be analyzed within 40 days of extraction.

7.0 <u>APPARATUS AND MATERIALS</u>

7.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for quantitation and confirmation:

7.1.1 RTX-35 30 meter, 0.53mm ID 0.83um film thickness, Restek Corporation Cat #10455

7.1.2 DB-1701 30 meter, 0.53mm ID 1.5um film thickness, J&W Scientific CAT #125-0732

7.2 Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

7.3 0.8 ml and 1.8 ml autosampler crimptop vials and caps.

7.4 Vial crimper

7.5 Borosilicate glass transfer pipets/transfer bulbs.

7.6 Safety glasses, non-powdered polyvinyl gloves, fume hood.

7.7 Properly cooled refrigerators separately for sample and standard storage.

7.8 Standard and Instrument Maintenance logbooks.

7.9 Sample extract refrigerator log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Solvents: All solvents should be pesticide grade or equivalent.

8.1.1 Hexane

8.1.2 Acetone

8.1.3 Toluene

8.1.4 Isooctane (2,2,4-trimethylpentane)

8.2 Standards

8.2.1 Calibration Stock Standard is prepared by diluting 1.0 ml of NSI Custom Pesticide Mixture Q-1132-01 to 100 mL in a pre-rinsed wolumetric flask with iso-octane. See Table 1 for final concentrations of compounds.

8.2.2 Single-component Calibration Standards at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard. The concentration should correspond to the expected range of concentrations found in actual samples and should bracket the linear range of the instrument. See Table 1 for final component concentrations.

8.2.3 Multicomponent Standards - A five point calibration curve is generated for multicomponent pesticide compounds Chlordane (technical) and Toxaphene when they are detected in a sample. Singel-point standards are analyzed after the single component pesticide calibration curves for identification purposes. For Multi-component Stock and Standard concentrations see Tables 2.0 & 2.1.

8.2.4 Surrogate Standard is prepared to monitor the performance of the extraction and analytical system. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked into each sample, blank and QC sample prior to extraction. (See Table 3 for spike concentrations).

8.2.5 Laboratory Control Sample (LCS) and Matrix Spiking Stock Standard Solution is prepared by diluting 1.0 ml of NSI Pesticide Mix Q-1065A-01 to 50 mL in a pre-rinsed volumetric flask with a 90% Iso-octane/10% Acetone mix. The working spike solution prepared for extractions is made by diluting 10 mL of the above stock solution to 100 mL in a pre-rinsed volumetric flas with an 80% Methanol/20% Aceton mix. 1.0 mL of the working solutioin is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the LCS sample. (See Table 4.)

8.2.5.1 If the batch of samples is for PCB only analysis, an MS/MSD of Aroclors only, is used. (See Table 4.1).

8.2.6 Instrument Breakdown Standard is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5).

8.3 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

8.4 All stock and working standards are stored in amber screw top bottles at 4 deg. C and replaced after 6 months or earlier if necessary.

8.5 All standard preparation must be documented in the Standards Logbook (Attachment I).

9.0 CALIBRATION

9.1 Retention Time (RT) windows are established by making three injections of the mid concentration standards throughout the course of a 72-hour period and calculating 3 x ± the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new column is installed.

9.1.1 RT windows are established as described above, however, the laboratory has established minimum RT windows as found in Table 8.

9.2 Calibration Standards

9.2.1 Initial calibration standards are analyzed by injecting 1 uL of each of the five levels of the single component calibration standard, and one level of all other multi-response pesticides/PCB's.

9.2.1.1 When analyzing for Aroclors only or when an Aroclor or multi-component pesticide is detected, an initial calibration consisting of five standard concentration levels is injected and the sample is re-analyzed against this five-point calibration curve. See Table 2.1 for Aroclor and multi-component pesticide curve standard concentrations.

9.2.2 The working calibration range of this method is defined by the initial calibration curve. All extracts with target compounds exceeding the curve must be diluted to within the calibration range.

9.3 The daily calibration verification is acceptable if all of the following are true.

9.3.1.1 The breakdown of Endrin or p'p'DDT is <= 15% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for p,p'DDT. If the breakdown does exceed 15% then corrective action must be taken prior to continuing the calibration verification.

$$\text{\% breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

% breakdown for Endrin =

$$\frac{\text{Endrin aldehyde peak area + Endrin Ketone peak area}}{\text{Total endrin peak area (Endrin+Endrin aldehyde+Endrin Ketone)}} \times 100$$

9.3.1.2 The midlevel calibration standard must have all compounds (+/-) 15% of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the midlevel calibration standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure.

9.3.1.3 An instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean.

9.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multicomponent targets must also be diluted so that the largest peak in the multicomponent does not exceed the response of the largest peak in the high level single-component calibration standard.

9.5 Calibration Verification (Calibration Check Standard - CCS)

9.5.1 A single midlevel standard (standard mix or multi-compound), is analyzed at a minimum of every 10 samples and at the end of the analysis sequence. The calibration factor for each compound to be quantitated must not exceed a 15% difference when compared to the average calibration factor from the calibration curve. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating. All samples that were injected prior to the standard exceeding the criteria must be reinjected if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

9.6 Calculations for water samples:

9.6.1 Quantitation of single-component target analytes is as follows:

$$\text{ug/L} = \frac{\text{Area of sample peak X Final volume of extract (uL) X Dilution Factor}}{\text{Avg Calibration Factor of Standard X Sample Volume extracted (mLs)}}$$

$$\text{CF} = \frac{\text{Peak area of the Standard}}{\text{Mass injected (ng)}}$$

9.6.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. Multi-component analytes are calculated by adding the areas of three to five of the largest peaks common to the sample and the standard and using this combined area in the following calculation:

$$\text{ug/L} = \frac{\text{(X)Final Volume of extract (uL) X Dilution Factor}}{\text{(Y)Sample volume extracted (mLs)}}$$

where:

X = the combined area of 3-5 peaks from sample.

Y = the average CF of the combined areas of 3-5 peaks from initial calibration standards.

9.7 Calculations for soil and oil samples:

9.7.1 The calculation for quantitation for single component target analytes is as follows:

$$\text{ug/kg} = \frac{\text{Area of sample peak X Final Volume of Extract (uL) X Dilution Factor}}{\text{Average CF of Standard X Sample volume extracted (g) X Decimal \% Solids}}$$

$$\text{CF} = \frac{\text{Peak Area of the Standard}}{\text{Mass injected (ng)}}$$

9.7.2 Multicomponent analytes are calculated by adding three to five of the largest peaks common to the sample and the standard, and using this combined area in the following calculation:

$$\text{ug/kg} = \frac{\text{(X)Final Volume of extract (uL) X Dilution Factor}}{\text{(Y)Sample volume extracted (g) X Decimal \% solids}}$$

where:

X = the combined area of 3-5 peaks from sample.

Y = the average CF of the combined areas of 3-5 peaks from initial calibration standards.

Note: Oils will not have a percent solids.

9.7.3 When more than one multicomponent is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen.

9.8 Corrective Action for Calibration

9.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

9.9 Corrective Action for Calibration Verification

9.9.1 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

9.9.2 Major corrective actions such as replacing the column or detector will require a new initial calibration.

9.9.3 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

10.0 QUALITY CONTROL

10.1 Refer to Table 6 for Practical Quantitation Limits (PQL's) for all compounds.

10.2 Matrix spike (MS), matrix spike duplicates (MSDs) and Laboratory Control Samples (LCS) are extracted within every batch of 20 samples or less. Recoveries must be within the limits listed in Table 7 of this SOP. If these criteria are not met, but the LCS data meet all of the recovery criteria, then the MS/MSD are documented as exhibiting suspected matrix interferences. If the LCS fails, check for instrument and/or column related problems and reanalyze the LCS. If the problem is corrected, the samples are reanalyzed. If these LCS still fails, the sample batch is reextracted and reanalyzed for an LCS compounds with failing recoveries below the lower acceptance range. If target compounds of interest have a recovery exceeding the highest acceptance range and the samples have no detected results for these target compounds, the data is reported with a discussion in the project narrative. In this instance, no quantitation limits or reported target compounds concentrations are affected.

10.3 Blanks

10.3.1 Frequency - method blanks must be analyzed with each batch of 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

10.3.2 Method blanks are spiked with surrogates,extracted and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 g of sodium sulfate.

10.3.3 Method blanks must contain less than the PQL for all the target compounds listed in Table 1.0.

10.3.4 All samples associated with an unacceptable method blank must be reextracted and reanalyzed.

10.4 Surrogates

10.4.1 The surrogates TCX and DCB are added to each sample, standard, blank and QC prior to extraction.

10.4.2 The advisory QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes. These limits are only advisory; therefore no further action is required if the criteria limits are not achieved

10.5 Calibration check sample	One per 24 hours and after every 10 samples
Breakdown standard	One per 24 hours
Instrument blank	One per 24 hours
MS/MSD	One pair every 20 samples
LCS	Every extraction batch (20 samples or less)
Surrogates	Every sample and standard and blank
Method blank (extracted)	Every extraction batch (20 samples or less)

10.6 Chain of Custody Record

10.6.1 All sampels are requested from the Sample Custodia using the Sample Request /Delivery Form (Attachment II).

10.6.2 When samples designated for Level III reports (or if specified by the client) are removed from storage for preparation or analysis they must be signed out utilizing the IEA internal chain of custody (COC). The samples shall then be signed back in on the internal COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation for analysis.

10.7 MDLs are determined in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Two HP5890 Series II Gas Chromatographs (GCs) with dual EC's and 7673A Twin Tower Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions are as follows:

a)

Front Injection port:	30 meter RTS-35 (See Section 7.1)
Column flow:	4.0 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:

Initial Temperature:	160 deg. C Hold 1.0 min.
Initial Ramp:	3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.:	15 degrees/min to 270 deg. C, Hold 16 min.

b)

Back Injection Port	DB-1701 (See Section 7.1)
Column flow:	5 ml/min He
Detector Make-up Flow:	65 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:

Initial Temperature:	160 deg. C, Hold 1.0 min.
Initial Ramp:	3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.:	15 degrees/min to 270 deg. C, Hold 16 min.

11.2 Soil samples are prepared in accordance with the SOP for SW-846 Method 3550 (Pesticide's/PCB's in Soil). IEA Doc# SPS01500.NC.

11.3 Water samples are prepared in accordance with the SOP for SW-846 Method 3510 (Pesticide's/PCB's in Water), IEA Doc# SPS01600.NC.

11.4 Sample Analysis

11.4.1 1.0 uL of extract is injected onto the GC column by an autosampler and the temperature program sequence is stared.

11.4.2 If a dilution is employed to bring a peak within the calibration range, both results are reported. The resulting change in quantitation limits and surrogate recovery must be reported for the diluted samples.

11.5 Qualitative Analysis

11.5.1 Target Compounds

11.5.1.1 The identification of a single component pesticide is based on the retention time data. The RT of a peak can be verified only from an on-scale chromatogram.

11.5.1.2 If a compound falls within the RT windows of the compound in the calibration curve, and is greater than the PQL, that sample would require confirmation by analysis on a second column.

11.6 Quantitative Analysis

11.6.1 Target Compounds

11.6.1.1 Target compounds are quantitated by the external standard technique using the peak area and the calibration factor determined during the initial calibration sequence.

11.6.1.2 When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

11.6.1.3 When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte to within the calibration range.

11.7 Data Acquisition

11.7.1 Data Acquisition and System Operation

11.7.1.1 Data is acquired from sample analyses using the Hewlett-Packard 1000 computer system with RTE operating system and LDS processing software. Analytical batches are set up using LDS with all the associated sample ID, dilution, and data file information. The analytical sequence setup mirrors the autosampler sequence setup. Automated post-acquisition quantitation is queued with the appropriate method file. The sequence is assigned using the LDS program and AS and starts acquiring when the A/D converter gets a start signal from the GC.

11.8 Manual integration Editing Flags

11.8.1 Manual integration shall be performed when the automated integration does not yield correctly u=integrated baselines. Manual integrations re flagged by the data system with the "FF" qualifier beside any manually integrated area. The analyst shall initial and date the hardcopy report for all manual integration performed.

12.0 CALCULATIONS

12.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass injected (ng)}}$$

12.2 Calculation of % Difference:

$$\%\ \text{Difference} = \frac{|\text{Calculated conc. - expected conc.}|}{\text{Expected conc.}} \times 100$$

12.3 Calculation of spike and QC check recoveries:

$$\%\ \text{Recoveries} = \frac{\text{Amount recovered}}{\text{Amount added}} \times 100\%$$

12.4 Calculation of Percent Moisture

$$\%\ \text{Moisture} = \frac{\text{Wet Sample (g) - Dry Sample (g)}}{\text{Wet Sample (g)}} \text{ X } 100$$

12.5 Calculation of Adjusted Practical Quantitation Limit

$$\text{Adjusted CRQL} = \frac{(\text{CRQL}) \text{ X } D_f}{D}$$

Where:

$$D = \frac{100 - \%\ \text{Moisture}}{100}$$

D_f = Dilution factor

12.6 Calculation for Target Compounds

See Sections 9.6 and 9.7.

13.0 ACCEPTANCE OF DATA

13.1 A calibration check standard is analyzed to verify the calibration curve by analysis of the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is (+/-) 15% of the expected value. The Calibration Check Standard is analyzed after every 10 samples.

13.2 Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is <= 15%.

13.3 The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 Method blanks are extracted with every batch of up to 20 samples or less to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.5 Matrix Spikes and Matrix Spike Duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 7.

13.6 Laboratory Control Samples are extracted with every batch of 20 samples. Acceptance criteria are listed in Table 7.

14.0 REPORTING OF RESULTS

14.1 All samples will have their target analytes reported in ug/L for waters and ug/Kg for soils. For an analyte to be reported, the concentration must be above the MDL. Results above the MDL and below the PQL will be flagged "J" as estimated, if requested by the client.

14.2 All results will be reported to two significant figures. Figures are rounded when the final concentration is achieved.

14.3 If it is determined that analytes are not present in the sample, the analytes will be reported as BQL (below quantitation limit).

14.4 The Quantitation Limit will be elevated proportionately in samples requiring dilutions.

14.5 Three different report levels are available:

14.5.1 A Level I data package includes: sample results, blank results and sampling COCs.

14.5.2 A Level II data package includes: Sample results, blank results, matrix spike results, matrix spike duplicate summary data, surrogate recovery data and sampling COCs.

14.5.3 A Level III data package includes: Sample results, blank results, matrix spike results, matrix spike duplicate summary data, project narrative, LCS results, surrogate recovery data, IEA assigned # (cross reference) index and sampling and internal COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Sulfur Removal, IEA Doc# GCS01500.NC.

15.2 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.3 SOP for SW-846 Method 3550 (Pesticide's/PCB's in Soil), IEA Doc# SPS01500.NC.

15.4 SOP for SW-846 Method 3510 (Pesticide's/PCB's in Water), IEA Doc# SPS01600.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 8081.

16.2 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 8000.

16.3 USEPA CLP OLMO3.1 Statement of Work.

17.0 SUBSTANTIVE REVISIONS

17.1 Draft Issue (12/10/96).

TABLE 1 - Single Component Calibration Concentrations, ug/mL

Analyte	Stock	Cal A	Cal B	Cal C	Cal D	Cal E
Aldrin	1.0	0.005	0.01	0.025	0.05	0.10
Dieldrin	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDT	6.0	0.03	0.06	0.15	0.30	0.60
p,p'DDE	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDD	6.0	0.03	0.06	0.15	0.30	0.60
Endosulfan I	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan II	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	6.0	0.03	0.06	0.15	0.30	0.60
Endrin	2.0	0.01	0.02	0.05	0.10	0.20
Endrin Aldehyde	6.0	0.03	0.06	0.15	0.30	0.60
Heptachlor	1.0	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	1.0	0.005	0.01	0.025	0.05	0.10
alpha-BHC	1.0	0.005	0.01	0.025	0.05	0.10
beta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
gamma-BHC	1.0	0.005	0.01	0.025	0.05	0.10
delta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
Methoxychlor	6.0	0.03	0.06	0.15	0.30	0.60
Endrin Ketone	6.0	0.03	0.06	0.15	0.30	0.60
TCX	1.0	0.005	0.01	0.025	0.05	0.10
DCB	1.0	0.005	0.01	0.025	0.05	0.10

TABLE 2 - Multi-Component Concentration, ug/mL

Multi-Component	Stock	Working Standard
AR 1221	10	0.20
AR 1232	10	0.10
AR 1242	10	0.10
AR 1248	10	0.10
AR 1254	10	0.10
Technical Chlordane	50.0	0.20
Toxaphene	10.0	0.50

TABLE 2.1
Multi-Component Concentration for 5-Point Calibrations, ug/mL

Compound	Stock	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
AR1221	10	0.25	0.50	1.0	2.0	4.0
AR1232	10	0.25	0.50	1.0	2.0	4.0
AR1242	10	0.25	0.50	1.0	2.0	4.0
AR1248	10	0.25	0.50	1.0	2.0	4.0
AR1254	10	0.25	0.50	1.0	2.0	4.0
AR1660	10	0.25	0.50	1.0	2.0	4.0
Toxaphene	10	0.25	0.50	1.0	1.5	2.0
Chlordane (Technical)	50	0.10	0.20	0.5	1.0	2.0

TABLE 3 - Surrogate Mix, ug/mL

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

Table 3.1
Surrogate Recovery Limits

Surrogate	Soil	Water
TCX	60-150	60-150
DCB	60-150	60-150

TABLE 4 - QC Check and Matrix Spike, ug/mL

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0

TABLE 4.1
Aroclor Matrix Spike Solution (ug/mL)

Compound	Stock	Std. Mix for Extractions (Added to each Aroclor MS)	Water Control Limits	Soil Control Limits
AR-1260	1000	10	8-127	8-127

TABLE 5 - Breakdown Std Conc., ug/mL

Compound	Stock (individual compound)	Standard (working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6 - Practical Quantitation Limits (PQL)

Analyte	PQLs for Waters (ug/mL)	PQLs for Soils (ug/kg)
alpha-BHC	0.05	8.0
beta-BHC	0.05	8.0
delta-BHC	0.05	8.0
gamma-BHC (Lindane)	0.05	8.0
Heptachlor	0.05	8.0
Aldrin	0.05	8.0
Heptachlor Epoxide	0.05	8.0
Endosulfan I	0.05	8.0
Dieldrin	0.10	16
p,p'DDE	0.10	16
Endrin	0.10	16
Endosulfan II	0.10	16
p,p'DDD	0.10	16
Endosulfar Sulfate	0.10	16
p,p'DDT	0.10	16
Methoxychlor	0.50	80
Toxaphene	1.0	160
AR 1016	1.0	80
AR 1221	2.0	80
AR 1232	1.0	80
AR 1242	1.0	80
AR 1248	1.0	80
AR 1254	1.0	160
AR 1260	1.0	160
Tech. Chlordane	0.20	80
Endrin Aldehyde	0.10	8.0
Endrin Ketone	0.10	8.0

Table 7
Matrix Spike
& Laboratory Control Sample Acceptance Criteria

Compound	Matrix Spike Recovery Limits (Soils)	Matrix Spike Recovery Limits (Waters)	LCS Recovery Limits (Soils)	LCS Recovery Limits (Waters)
alpha-BHC	37-134	37-134	43-105	41-126
gamma-BHC	32-127	32-127	52-108	60-127
Heptachlor	34-111	34-111	52-111	61-111
Aldrin	42-122	42-122	42-122	64-113
beta-BHC	17-147	17-147	63-125	17-147
delta-BHC	19-140	19-140	19-140	19-140
Heptachlor epoxide	37-142	37-142	54-121	76-128
Endosulfan I	45-153	45-153	66-138	87-143
4,4'-DDE	30-145	30-145	61-143	46-145
Dieldrin	36-146	36-146	57-146	75-146
Endrin	30-147	30-147	55-126	58-133
4,4"-DDD	31-141	31-141	53-127	69-141
Endosulfan II	10-200	10-200	47-121	53-153
4,4'-DDT	25-160	25-160	25-160	46-134
Endrin Aldehyde	10-200	10-200	10-200	10-200
Methoxychlor	25-160	25-160	63-133	47-142
Endosulfan sulfate	26-144	26-144	26-144	26-144
Endrin Ketone	10-200	10-200	10-200	10-200

Table 8
Minimum Retention Time (RT) Windows

Target Compound	RT Window Minimum
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07
Heptachlor epoxide	±0.07
Dieldrin	±0.07
Endrin	±0.07
Endrin aldehyde	±0.07
Endrin ketone	±0.07
DDD	±0.07
DDE	±0.07
DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

ATTACHMENT I

STANDARDS LOGBOOK

Standard Name: ______________
Location: ______________

Lot Code #: ______________
Solvent Used: ______________
Volume of Solvent Used: ______________
Prepared By: ______________
Date Prepared: ______________
Expiration Date: ______________

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc	Amt Added	Final Vol
1.						
2.						
3.						
4.						
5.						

Calculations:

Analyte	Final Analyte Concentration	Analyte	Final Analyte Concentration

Comments: ______________

Secondary Review By: ______________ Date: ______________

ATTACHMENT II

Sample Request/Delivery Form

Date:________________ Department Name:______________________________ COCs Received by______________________

#	Requestor	Project #	Sample #	Location	QC Level					Out	In	Matrix*
					I	II	III	CLP	COC			
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												

Matrix*: L = Leachate S = Soil W = Water O = Other

6D

SW-846 PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: INDUSTRIAL & ENVIRONMENTAL ANALYSTS

Lab Code: IEA Case No.: 2182-016 Method: 8080 SDG No.: 11245

Instrument ID: HP5890P6 GC Column:DB-1701

ID: 0.53 (mm) Date(s) Analyzed: 11/15/96

Level (x lvl1): lvl1:1.0 lvl2:2.0 lvl3:5.0 lvl4:10 lvl5:20

COMPOUND	RT OF STANDARDS LVL1	LVL2	LVL3	LVL4	LVL5	CURVE TYPE *
alpha-BHC	13.67	13.66	13.67	13.67	13.67	Averaged
beta-BHC	19.15	19.15	19.16	19.16	19.15	Averaged
delta-BHC	20.19	20.19	20.19	20.19	20.18	Averaged
gamma-BHC (Lindane)	15.55	15.55	15.55	15.55	15.55	Averaged
Heptachlor	16.46	16.45	16.46	16.46	16.46	Averaged
Aldrin	17.70	17.70	17.70	17.70	17.70	Averaged
Heptachlor epoxide	20.83	20.82	20.83	20.83	20.82	Averaged
Endosulfan I	21.95	21.95	21.95	21.95	21.95	Averaged
Dieldrin	23.43	23.43	23.43	23.43	23.43	Averaged
4,4'-DDE	22.89	22.89	22.89	22.89	22.89	Averaged
Endrin	24.17	24.17	24.17	24.17	24.17	Averaged
4,4'-DDD	25.81	25.80	25.81	25.81	25.83	Averaged
Endosulfan II	25.95	25.94	25.95	25.95	25.95	Averaged
4,4'-DDT	26.35	26.35	26.36	26.36	26.38	Averaged
Endo sulf/Methoxy	28.38	28.37	28.38	28.38	28.41	Averaged
Endrin ketone	29.66	29.66	29.66	29.66	29.68	Averaged
Endrin aldehyde	27.36	27.36	27.36	27.36	27.36	Averaged
Tetrachloro-m-xylene	9.29	9.29	9.29	9.29	9.29	Averaged
Decachlorobiphenyl	32.83	32.84	32.84	32.84	32.84	Averaged

* Origin included in linear, quadratic, and averaged curves

6D

SW-846 PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: INDUSTRIAL & ENVIRONMENTAL ANALYSTS

Lab Code: IEA   Case No.: 2182-016   Method: 8080   SDG No.: 11245

Instrument ID: HP5890P5   GC Column:RTX-35

ID: 0.53 (mm)   Date(s) Analyzed: 11/15/96

Level (x lvl1): lvl1:1.0 lvl2:2.0 lvl3:5.0 lvl4:10 lvl5:20

COMPOUND	RT OF STANDARDS LVL1	LVL2	LVL3	LVL4	LVL5	CURVE TYPE *
alpha-BHC	14.32	14.32	14.32	14.31	14.31	Averaged
beta-BHC	16.63	16.62	16.62	16.62	16.62	Averaged
delta-BHC	18.29	18.29	18.29	18.29	18.28	Averaged
gamma-BHC (Lindane)	16.19	16.19	16.19	16.18	16.18	Averaged
Heptachlor	18.03	18.03	18.03	18.03	18.02	Averaged
Aldrin	19.56	19.56	19.56	19.55	19.55	Averaged
Heptachlor epoxide	22.07	22.06	22.06	22.06	22.06	Averaged
Endosulfan I	23.53	23.53	23.52	23.52	23.52	Averaged
Dieldrin	24.67	24.67	24.67	24.66	24.66	Averaged
4,4'-DDE	24.43	24.44	24.43	24.43	24.43	Averaged
Endrin	25.89	25.89	25.89	25.88	25.88	Averaged
Endosulfan II	26.55	26.55	26.55	26.54	26.54	Averaged
4,4'-DDD	26.40	26.40	26.40	26.40	26.40	Averaged
Endosulfan sulfate	28.12	28.12	28.12	28.12	28.12	Averaged
4,4'-DDT	27.45	27.45	27.45	27.45	27.44	Averaged
Methoxychlor	30.13	30.12	30.12	30.12	30.12	Averaged
Endrin ketone	30.51	30.51	30.51	30.50	30.50	Averaged
Endrin aldehyde	27.58	27.58	27.58	27.57	27.57	Averaged
Tetrachloro-m-xylene	11.06	11.06	11.06	11.05	11.05	Averaged
Decachlorobiphenyl	37.48	37.49	37.48	37.48	37.47	Averaged

* Origin included in linear, quadratic, and averaged curves

IEA Pesticide Standard Report

Sample Name : AR1660C/POC96-128 Report No : 2.00
esult File : /RESULT/P8091096_002.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 1814 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 2 Bottle no. : 2

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.032	46811	VV	0.00000	
2	.63		.029	1975	VV	0.00000	
3	.87		.045	2980	PV	0.00000	
4	.96		.065	481	VB	0.00000	
5	1.14		.018	166	BB	0.00000	
6	1.83		.052	782	BV	0.00000	
7	2.27		.039	1222	PV	0.00000	
8	2.32		.049	1469	VV	0.00000	
9	2.90		.058	2610	VV	0.00000	
10	3.00		.058	1286	VV	0.00000	
11	3.17		.061	1421	PV	0.00000	
12	3.69		.078	9184	BV	0.00000	
13	4.61	4.62	.086	141818	BV	.20221	Tetrachloro-m-xylene
14	5.02		.044	496	VV	0.00000	
15	5.12		.070	4116	VV	0.00000	
16	5.23		.101	26621	VV	0.00000	
17	5.74		.130	31474	VV	0.00000	
18	6.21		.099	29963	PV	0.00000	
19	6.39		.130	186112	VV	0.00000	
20	7.00		.098	27956	VV	0.00000	
21	7.59		.116	410244	PV	0.00000	
22	7.95		.115	32380	VV	0.00000	
23	8.22		.117	112708	VV	0.00000	
24	8.35		.127	178739	VV	0.00000	
25	8.83		.145	96751	VV	0.00000	
26	9.11		.142	657197	VV	0.00000	
27	9.52		.192	373404	VV	0.00000	AR1016
28	9.86		.148	249127	VV	0.00000	
29	10.26		.207	635955	VV	0.00000	
30	11.04		.145	343425	VV	0.00000	
31	11.36		.192	314948	VV	0.00000	
32	11.93		.162	64320	VV	0.00000	
33	12.14		.206	43846	VV	0.00000	
34	12.51		.193	260697	VV	0.00000	
35	13.10		.171	32192	VV	0.00000	
36	13.38		.182	208548	VV	0.00000	

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
37	13.63		.172	29840	VB	0.00000	
38	14.66		.182	6116	PV	0.00000	
39	15.29		.232	126190	PV	0.00000	
40	15.82		.238	339452	VV	0.00000	
41	16.22		.264	132842	VV	0.00000	
42	16.80		.255	557041	VV	0.00000	
43	17.60		.297	73758	VV	0.00000	
44	18.10		.286	90211	VV	0.00000	
45	18.64		.310	627731	VV	0.00000	
46	19.46		.431	360944	VV	0.00000	
47	20.08		.383	281006	VV	0.00000	
48	21.70		.429	742981	PV	0.00000	
49	22.12 (1)		.310	428849	VV	0.00000	
50	22.50		.328	256551	VV	0.00000	
51	23.71		.380	71823	PV	0.00000	
52	24.58 (2)		.255	419084	VV	0.00000	
53	24.93		.255	231035	VV	0.00000	
54	25.22		.239	111202	VV	0.00000	
55	26.21		.235	81848	PV	0.00000	
56	26.71 (3)		.264	889567	VV	0.00000	
57	28.54		.128	102143	PV	0.00000	
58	28.81 (4)		.243	648658	VV	0.00000	
59	29.66		.129	5242	PV	0.00000	
60	30.76		.131	51642	PV	0.00000	
61	32.18		.138	131141	PV	0.00000	
62	33.45		.033	9606	PV	0.00000	
63	34.07	#34.09	.165	161184	PV	.21206	Decachlorobiphenyl

AR1260

$\frac{2386158}{2511227}$ 0.95

5.0%D

Total Area : 11501114 Total PPB : .414

Report Time : 1905 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_002.RES

IEA Pesticide Standard Report

Sample Name : AR1242C/POC96-133 Report No : 3.00
Result File : /RESULT/P8091096_003.RES
Column Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890PB
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 1911 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 3 Bottle no. : 3

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.031	44861	VV	0.00000	
2	.63		.030	2887	VV	0.00000	
3	.78		.028	308	PV	0.00000	
4	.87		.052	2476	VV	0.00000	
5	1.01		.109	2307	VV	0.00000	
6	1.14		.069	1238	PV	0.00000	
7	1.27		.114	487	VB	0.00000	
8	2.27		.047	1469	BV	0.00000	
9	2.31		.045	1362	VV	0.00000	
10	2.89		.068	2739	VV	0.00000	
11	2.99		.052	1025	VV	0.00000	
12	3.17		.067	1033	PV	0.00000	
13	3.69		.078	6856	VV	0.00000	
14	4.61	4.62	.087	150953	PV	.21523	Tetrachloro-m-xylene
15	5.12		.073	3948	VV	0.00000	
16	5.23		.101	23064	VV	0.00000	
17	5.74		.130	26276	VV	0.00000	
18	6.20		.098	24926	PV	0.00000	
19	6.39 (1)		.135	158594	VV	0.00000	
20	7.00		.101	23657	VB	0.00000	
21	7.59 (2)		.116	333530	BV	0.00000	
22	7.94		.114	25526	VV	0.00000	
23	8.21		.117	88620	VV	0.00000	
24	8.35		.129	146881	VV	0.00000	
25	8.83		.150	78766	VV	0.00000	
26	9.11 (3)		.149	522668	VV	0.00000	
27	9.52		.201	308339	VV	0.00000	
28	9.85		.154	203053	VV	0.00000	
29	10.25 (4)		.209	522237	VV	0.00000	
30	11.04		.149	297425	VV	0.00000	
31	11.35		.212	330180	VV	0.00000	
32	11.92		.156	82257	VV	0.00000	
33	12.14		.209	148034	VV	0.00000	
34	12.60		.300	480848	VV	0.00000	
35	13.08		.205	53893	VV	0.00000	

$\frac{1537029}{1413265}$ 1.09

8.89, D

AR1242

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	13.64		.437	382891	VV	0.00000	AR1242 (cont.)
37	14.65		.297	70015	VV	0.00000	
38	15.20		.369	97373	VV	0.00000	
39	15.79		.343	111260	VV	0.00000	
40	16.78		.434	67533	VV	0.00000	
41	17.58		.485	83612	VV	0.00000	
42	18.64		.459	19932	VV	0.00000	
43	20.35		.424	35538	VV	0.00000	
44	21.72		.283	9418	PV	0.00000	
45	34.07		4.969	-4673524	PB	0.00000	

Total Area : 4980297 Total PPB : .215

Report Time : 2001 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_003.RES

IEA Pesticide Standard Report

Sample Name : AR1248C/POC96-138 Report No : 4.00
Result File : /RESULT/P8091096_004.RES
Column Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890PB
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2008 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 4 Bottle no. : 4

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.031	52814	VV	0.00000	
2	.63		.034	2840	VV	0.00000	
3	.78		.017	186	PV	0.00000	
4	.87		.041	2313	VB	0.00000	
5	1.14		.054	958	PV	0.00000	
6	1.20		.021	55	VV	0.00000	
7	2.27		.040	1158	BV	0.00000	
8	2.31		.049	1374	VV	0.00000	
9	2.90		.066	2545	PV	0.00000	
10	2.99		.047	1148	VV	0.00000	
11	3.17		.052	1076	VV	0.00000	
12	3.69		.079	5136	BV	0.00000	
13	4.61	4.62	.087	146153	PV	.20839	Tetrachloro-m-xylene
14	5.22		.122	2473	VV	0.00000	
15	5.63		.049	572	BV	0.00000	
16	6.24		.106	1948	BV	0.00000	
17	6.39		.114	13769	VV	0.00000	
18	7.00		.093	6101	BV	0.00000	
19	7.59 (1)		.114	166143	BV	0.00000	
20	7.94		.140	10484	VV	0.00000	
21	8.21		.103	37764	VV	0.00000	
22	8.35		.131	56318	VV	0.00000	
23	8.82		.175	30124	VV	0.00000	
24	9.10 (2)		.151	328063	VV	0.00000	2118074 / 1961573
25	9.51		.235	206291	VV	0.00000	
26	9.85		.147	145601	VV	0.00000	
27	10.25 (3)		.197	766862	VV	0.00000	
28	11.03		.141	453878	VV	0.00000	
29	11.35		.179	435698	VV	0.00000	1.08
30	11.92		.147	105294	VV	0.00000	
31	12.14		.195	220538	VV	0.00000	
32	12.59 (4)		.290	857006	VV	0.00000	8.0%D
33	13.09		.197	83672	VV	0.00000	AR1248
34	13.64		.429	708447	VV	0.00000	
35	14.35		.223	45069	VV	0.00000	

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	14.65		.258	136904	VV	0.00000	
37	15.19		.335	213777	VV	0.00000	
38	15.79		.291	253507	VV	0.00000	
39	16.77		.373	131162	VV	0.00000	AR1248 (Cont)
40	17.58		.432	182718	VV	0.00000	
41	18.63		.423	33395	VV	0.00000	
42	19.49		.292	14252	VV	0.00000	
43	20.34		.414	96597	PV	0.00000	
44	21.71		.335	27236	VV	0.00000	
45	22.48		.217	1727	PV	0.00000	
46	24.55		.285	-7954	PV	0.00000	
47	26.74		1.831	-22590	PV	0.00000	
48	34.07		1.154	-1114806	PB	0.00000	

Total Area : 5991147 Total PPB : .208

Report Time : 2100 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_004.RES

IEA Pesticide Standard Report

Sample Name : AR1254C/POC96-143 Report No : 5.00
esult File : /RESULT/P8091096_005.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2105 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 5 Bottle no. : 5

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.038	31260	VV	0.00000	
2	.63		.030	1737	VV	0.00000	
3	.78		.038	718	PV	0.00000	
4	.87		.064	4169	VV	0.00000	
5	.96		.054	1518	VV	0.00000	
6	1.01		.090	2380	VV	0.00000	
7	1.14		.107	4456	VV	0.00000	
8	1.32		.140	4526	VV	0.00000	
9	1.39		.070	2064	VV	0.00000	
10	1.71		.315	7585	VV	0.00000	
11	1.83		.101	3539	VV	0.00000	
12	1.97		.120	2439	VV	0.00000	
13	2.27		.118	5271	VV	0.00000	
14	2.89		.060	1885	BV	0.00000	
15	3.17		.070	1031	PV	0.00000	
16	3.69		.077	4956	BB	0.00000	
17	4.61	4.62	.087	148200	BV	.21131	Tetrachloro-m-xylene
18	5.62		.097	1759	VV	0.00000	
19	6.39		.142	2826	BV	0.00000	
20	7.59		.103	7268	BB	0.00000	
21	8.22		.096	1746	BV	0.00000	
22	8.36		.130	2728	VB	0.00000	
23	8.92		.145	2988	BV	0.00000	
24	9.11		.171	15262	VV	0.00000	
25	9.42		.120	7497	VV	0.00000	
26	9.49		.183	10323	VV	0.00000	
27	9.85		.165	8304	VV	0.00000	
28	10.22		.159	338706	VV	0.00000	AR1254
29	11.03		.137	142392	VV	0.00000	
30	11.35		.197	82404	VV	0.00000	
31	11.92		.179	19915	VV	0.00000	
32	12.15		.189	60601	VV	0.00000	
33	12.51		.219	671475	VV	0.00000	
34	13.10		.175	120430	VV	0.00000	
35	13.38		.232	662577	VV	0.00000	

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	13.65		.204	289417	VV	0.00000	AR1254 (cont)
37	14.34		.206	44953	VV	0.00000	
38	14.65		.224	220692	VV	0.00000	
39	15.17		.294	457916	VV	0.00000	
40	15.79 2		.244	703992	VV	0.00000	
41	16.18		.247	108848	VV	0.00000	
42	16.78		.293	396633	VV	0.00000	
43	17.58 3		.335	468596	VV	0.00000	
44	18.07		.292	107120	VV	0.00000	1.03
45	18.63		.329	335085	VV	0.00000	2480675 / 2408912
46	19.49		.301	199356	VV	0.00000	
47	20.33		.504	304477	VV	0.00000	3.0%
48	21.12		.252	31788	VV	0.00000	
49	21.70 4		.414	645510	VV	0.00000	
50	22.45		.342	71113	VB	0.00000	
51	24.53		.351	204689	BV	0.00000	
52	25.22		.224	13029	VV	0.00000	
53	26.18		.246	59760	PV	0.00000	
54	26.70		.270	79869	VV	0.00000	
55	28.81		.119	26973	PV	0.00000	
56	34.07		.306	-306185	PB	0.00000	~~Tex~~ DCB

Total Area : 7156751 Total PPB : .211

Report Time : 2204 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_005.RES

IEA Pesticide Standard Report

Sample Name : AR1221C/POC96-311 Report No : 7.00
Result File : /RESULT/P8091096_007.RES
Column Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2259 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 7 Bottle no. : 7

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.052	42420	BV	0.00000	
2	.79		.068	3551	PV	0.00000	
3	.89		.057	2057	VV	0.00000	
4	.95		.049	11572	VV	0.00000	
5	1.03		.109	21753	VV	0.00000	
6	1.32		.094	2098	PV	0.00000	
7	1.49		.200	3484	VV	0.00000	
8	1.55		.045	382	VV	0.00000	
9	1.69		.090	397	VB	0.00000	
10	1.92		.070	2155	BB	0.00000	
11	2.31		.065	2137	BB	0.00000	
12	2.76		.096	2698	BV	0.00000	
13	2.90		.094	1664	VV	0.00000	
14	2.99		.067	639	VB	0.00000	
15	3.67		.092	406317	BV	0.00000	
16	4.26		.070	960	VV	0.00000	
17	4.61	4.62	.080	366844	BV	.51857	Tetrachloro-m-xylene
18	4.78		.135	172579	VV	0.00000	
19	5.12		.079	64584	VV	0.00000	
20	5.22		.112	163873	VV	0.00000	
21	5.73		.124	423621	VV	0.00000	
22	6.20		.103	254557	VV	0.00000	
23	6.39		.136	1076501	VV	0.00000	
24	7.00		.190	59386	VV	0.00000	
25	7.59		.124	285687	VV	0.00000	
26	7.82		.182	86378	VV	0.00000	
27	8.09		.122	105533	VV	0.00000	
28	8.21		.110	96192	VV	0.00000	
29	8.35		.151	152670	VV	0.00000	
30	8.83		.161	74568	VV	0.00000	
31	9.11		.157	349695	VV	0.00000	
32	9.52		.210	204362	VV	0.00000	
33	9.86		.168	129320	VV	0.00000	
34	10.26		.230	279513	VV	0.00000	
35	11.04		.156	138171	VV	0.00000	

AR1221

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	11.36		.238	156260	VV	0.00000	AR1221 (cont.)
37	11.93		.165	38943	VV	0.00000	
38	12.14		.222	66629	VV	0.00000	
39	12.60		.326	214669	VV	0.00000	
40	13.09		.190	24993	VV	0.00000	
41	13.65		.475	163679	VV	0.00000	
42	14.65		.295	32681	VV	0.00000	
43	15.20		.392	41352	VV	0.00000	
44	15.79		.336	42215	VV	0.00000	
45	16.80		.416	20801	VV	0.00000	
46	17.59		.406	20750	VV	0.00000	
47	20.32		.410	9772	PV	0.00000	
48	21.73		.179	2535	PV	0.00000	
49	34.07		1.239	-2697185	PB	0.00000	

Total Area : 5823594 Total PPB : .519

Report Time : 2349 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_007.RES

IEA Pesticide Standard Report

Sample Name : AR1232C/POC96-312 Report No : 8.00
Result File : /RESULT/P8091096_008.RES
Column Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890PB
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2356 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 8 Bottle no. : 8

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.052	38121	BV	0.00000	
2	.79		.065	3408	PV	0.00000	
3	.89		.056	1683	VV	0.00000	
4	.95		.048	11600	VV	0.00000	
5	1.03		.108	21216	VV	0.00000	
6	1.34		.102	2266	PV	0.00000	
7	1.42		.086	1811	VV	0.00000	
8	1.50		.098	1542	VV	0.00000	
9	1.61		.060	327	VV	0.00000	
10	1.69		.077	237	VV	0.00000	
11	1.93		.069	1698	BB	0.00000	
12	2.31		.079	3321	BV	0.00000	
13	2.74		.145	3370	PV	0.00000	
14	2.90		.077	1062	VV	0.00000	
15	3.00		.062	368	VV	0.00000	
16	3.67		.095	130913	VV	0.00000	
17	4.11		.069	612	VB	0.00000	
18	4.61	4.62	.081	379783	PV	.53686	Tetrachloro-m-xylene
19	4.78		.148	71899	VV	0.00000	
20	5.12		.080	23231	VV	0.00000	
21	5.23		.114	77548	VV	0.00000	
22	5.74		.130	153014	VV	0.00000	
23	6.20		.104	104184	VV	0.00000	
24	6.39		.142	533284	VV	0.00000	
25	7.00		.159	60060	VV	0.00000	
26	7.59		.117	450735	VV	0.00000	
27	7.82		.090	31788	VV	0.00000	
28	7.94		.109	42245	VV	0.00000	
29	8.10		.093	45730	VV	0.00000	
30	8.21		.111	133416	VV	0.00000	AR1232
31	8.35		..142	232854	VV	0.00000	
32	8.83		.151	116881	VV	0.00000	
33	9.11		.151	744635	VV	0.00000	
34	9.52		.202	429069	VV	0.00000	
35	9.86		.158	284548	VV	0.00000	

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	10.26		.213	654399	VV	0.00000	
37	11.04		.148	381490	VV	0.00000	
38	11.36		.214	409967	VV	0.00000	
39	11.93		.156	103707	VV	0.00000	AR1232
40	12.14		.208	169595	VV	0.00000	(cont.)
41	12.61		.297	580569	VV	0.00000	
42	13.08		.200	60885	VV	0.00000	
43	13.65		.416	425152	VV	0.00000	
44	14.65		.293	68586	VV	0.00000	
45	15.20		.368	93417	VV	0.00000	
46	15.79		.339	109638	VV	0.00000	
47	16.78		.389	69985	VV	0.00000	
48	17.58		.503	67993	VV	0.00000	
49	18.64		.396	24307	VV	0.00000	
50	19.48		.456	12041	VV	0.00000	
51	20.34		.460	28246	VV	0.00000	
52	21.72		.347	12633	PV	0.00000	
53	22.09		.263	6355	VV	0.00000	
54	22.50		.258	2751	VV	0.00000	
55	24.58		.132	4523	PV	0.00000	
56	24.94		.258	4939	VV	0.00000	
57	25.25		.229	1284	VV	0.00000	
58	26.71		.122	7931	PV	0.00000	
59	28.82		.121	-6203	PV	0.00000	
60	34.07		.022	-49379	PB	0.00000	

otal Area : 7438857 Total PPB : .537

Report Time : 0046 11Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_008.RES

15. IEA SOP FOR SULFUR REMOVAL FROM SOLVENT EXTRACTS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director ____________________

Quality Assurance Manager ____________________

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for removing sulfur from pesticide extracts.

3.0 SUMMARY OF METHOD

3.1 This method outlines the procedure for the removal of sulfur from a pesticide extract. Transfer the extract to a 40 ml vial and add TBA-Sulfite and 2-propanol. Then add water , shake, and remove hexane layer for analysis.

3.2 This SOP is based on the following method:

- USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) 2/88

4.0 INTERFERENCES

4.1 - Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

5.0 SAFETY

5.1 All compounds listed in Section 8 are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

* Clear 40 ml VOA vials with Teflon-coated liner.

* Sample vials are never cleaned and reused.

7.0 APPARATUS AND MATERIALS

7.1
* 40 ml VOA vials with teflon-lined screw caps
* 0.8 ml and 1.8 ml autosampler crimptop vials and caps.
* Vial crimper
* Sterile Borosilicate glass transfer pipets/transfer bulbs.
* Safety glasses, non-powdered polyvinyl gloves, fume hood.
* Properly cooled refrigerators each for sample storage.
* Standard and Instrument Maintenance logbooks.
* Sample log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 **Solvents:** Hexane and 2-propanol should be pesticide grade or equivalent.

8.2 TBA-sulfite reagent: Using certified A.C.S. grade anhydrous Sodium Sulfite and Tetrabutylammonoium hydrogen sulfate, 97%, prepare as follows:

Dissolve 3.4 g of TBA into 100.0ml DI water and mix well. Rinse 3 times with 20 ml of hexane and discard hexane. Add 25 g of Sodium Sulfite, mix well, then add additional Sodium Sulfite until some remains undissolved.

8.3 Deionized water.

9.0 CALIBRATION

Not Applicable

10.0 QUALITY CONTROL

10.1 If only a partial set of extracts needs sulfur cleanup, set up a new reagent blank with 2 ml of hexane.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Transfer 2 ml of extract to a 40 ml VOA vial. Add 2 ml TBA-silfite reagent and 2 ml 2-propanol , cap the vial, and shake for at least 1 min. If the extract is colorless or if the initial color is unchanged, and if clear crystals are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100 mg portions until a solid residue remains after shaking.

11.2 Add 5 ml DI water and shake for at least 1 min. Allow the extract to stand for 5-10 min. and use the hexane(top) layer for analysis.

12.0 CALCULATIONS

Not applicable

13.0 ACCEPTANCE OF DATA

13.1 Method or Reagent blank must contain no target compounds at a level above the quantitation limits of the analysis method.

14.0 REPORTING OF RESULTS

Not applicable

15.0 SUPPLEMENTAL DOCUMENTS

Not applicable

16.0 REFERENCES

16.1 Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 2/88.

17.0 SUBSTANTIVE REVISIONS

Not applicable

16. IEA SOP FOR SW-846 8150 HERBICIDES IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method outlines the gas chromatographic procedure for detecting and determining the concentration of chlorinated phenoxy acid herbicides in environmental water samples.

2.2 See Table 1 for Target Compounds.

2.3 The following test code is addressed in the SOP:

GCH_19

3.0 SUMMARY OF METHOD

3.1 This method defines the steps for analyzing environmental water samples for chlorinated phenoxy acid herbicides. The samples are acidified, then extracted to remove the herbicide residues from the environmental matrix. The residue is hydrolyzed to an acid and solvent washed to remove extraneous material. The acid is then derivitized to methyl esters which are analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

-EPA SW-846 Method 8150 (Chlorinated Herbicides)
-EPA SW-846 Method 8000 (Gas Chromatography)

3.3 This SOP deviates from the method as follows:

3.3.1 Capillary columns are used instead of packed columns.

4.0 INTERFERENCES

4.1 Organic acids, especially chlorinated acids, cause the most direct interference with this method. Phenols, including chlorophenols, may also interfere with this procedure.

4.2 Phthalate esters can interfere with herbicide determination; avoid any contact with plastics to best minimize this problem.

4.3 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during extraction or analysis. Glassware, glass wool and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this problem.

5.0 SAFETY

5.1 All compounds listed in Tables 1 - 3 (see attached documents) along with their respective solvents (i.e., hexane, iso-octane, acetone, toluene) and the derivitizing agents used in this method are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these materials. All work with these chemicals should be performed in a fume hood.

5.2 Material Safety Data Sheets (MSDS) for all chemicals used in this procedure are present in the laboratory for immediate access. Specific MSDS should be reviewed for a complete knowledge of the toxicity and precaution of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Water samples are collected with 2x1 liter amber glass containers with Teflon-coated liners.

6.1.2 Certified bottleware is purchased, precleaned.

6.1.3 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers and kept at 4 degrees C for transport to the laboratory. Samples should arrive at the laboratory within 24 hours of sample collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 degrees C.

6.4 Holding Times

6.4.1 All samples and extracts are stored at 4 degrees C.

6.4.2 Water samples must be extracted within 7 days from sampling.

6.4.3 All extracts must be analyzed within 40 days from the date of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for quantitation and confirmation:

7.1.1 DB-608 30 meter
0.053 mm ID 0.83 um film thickness
J&W Scientific CAT #125-1730 or equivalent (i.e., Restek RTX-35).

7.1.2 DB-5 30 meter
0.53 mm ID 1.5 um film thickness
J&W Scientific CAT #125-5032 or equivalent (i.e., Restek RTX-5).

7.1.3 DB-1701 30 meter
0.53 mm ID 1.0 um film thickness
J&W Scientific CAT #125-0732.

7.2 Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs.

7.3 0.8 mL and 1.8 mL autosampler crimptop vials and caps.

7.4 Vial crimper.

7.5 Sterile Borosillicate glass transfer pipets/transfer bulbs.

7.6 Safety glasses, non-powdered polyvinyl gloves, fume hood.

7.7 Properly cooled refrigerators each for sample, extract and standard storage.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Solvents: Hexane, Acetone, Toluene, Iso-octane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

8.1.1 Hexane and Acetone must be approved through the IEA Solvent Approval Program according to the SOP for Solvent/Chemical Reagent Approval, IEA Doc #QAS00401.NET.

8.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer for purity and content. Certificates for stock standards must be maintained on file in the laboratory. All standards received are documented in the Stock Standard Receipt Logbook (Attachment III).

8.3 All stock and working standards are stored in amber screw top bottles at 4 degrees C and replaced after 6 months or earlier if necessary.

8.4 Calibration stock standard for the extracted herbicide standard is prepared by adding the following to a 100 mL volumetric flask then bringing up to volume with acetone.

2.0 mL 2,4-D (2,4-Dichlorophenoxyacetic Acid) at 1000 ug/mL
400 uL 2,4,5-T (2,4,5-Trichlorophenoxyacetic Acid) at 500 ug/mL
200 uL Silvex (2-(2,4,5-Trichlorophenoxy)propionic Acid) at 1000 ug/mL
0.5 mL DCAA (2,4-Dichlorophenylacetic acid) at 1000 ug/mL
200 uL PCP (Pentachlorophenol) at 1000 ug/mL

These solutions are purchased, precertified, from Accustandard, Inc. See Table 1 for final concentrations.

8.4.1 A herbicide calibration standard is prepared by adding 1.0 mL of the Herbicide standard solution (Section 8.4) to 1.0 L of acidified reagent water. This standard goes through the extraction and esterification process with each batch of samples. See SOP for Extraction of Herbicides in Water for Method 8150, IEA Doc #SPS01001.NC for details of the extraction and esterification procedures.

8.5 Single-component Calibration Standards, at a minimum of 5 concentration levels, are prepared through dilution of the extracted herbicide standard. One concentration level should be near but above the method detection limit. The remaining concentrations should correspond to the linear range of the instrument. See Table 4 for final component concentrations.

8.6 Surrogate standard is prepared to monitor the performance of the extraction and analytical system. Samples, blanks and spikes all have surrogate solution spiked prior to extraction. The herbicide surrogate solution is prepared by adding 0.5 mL of DCAA at 1000 ug/mL to a 100 mL volumetric flask and bringing this to volume with acetone for a final concentration of 5.0 ug/mL.

8.7 Matrix Spike solution for the extracted herbicide matrix spike and matrix spike duplicate is prepared by adding the following to a 100 mL volumetric flask then bringing to volume with acetone.

400 uL 2,4-D (2,4-Dichlorophenoxyacetic Acid) at 1000 ug/mL
400 uL 2,4,5-T (2,4,5-Trichlorophenoxyacetic Acid) at 500 ug/mL
40 uL Silvex (2-(2,4,5-Trichlorophenoxy)propionic Acid at 1000 ug/mL

200 uL PCP (Pentachlorophenol) at 1000 ug/mL

These solutions are purchased, precertified, from Accustandard, Inc.

8.8 A Laboratory Control Sample (LCS) standard is prepared containing the target compounds. The LCS must be prepared from a standard source that is a different vendor or stock standard lot than the standards used in calibration. The concentration of the LCS for this procedure is found in Section 8.7. The LCS preparation is as found in Section 8.7.

8.9 All standard preparations are documented in the Standard Preparation Logbook (Attachment I).

9.0 CALIBRATION

9.1 Analyze the 5 concentration levels of standards in the order of low to high to develop the initial calibration. Using an external standard procedure, verify the calibration response factors (RF's) as generated for each compound and the linearity (%RSD) of each.

9.1.1 If the %RSD is <20%, linearity through the origin is assumed and an average response factor can be used for concentration calculations.

9.1.2 If linearity does not meet 20%, then linear curves are generated for each component using the method calibration function within the HP3350 software. The curves are prepared based on an origin included first order linear regression. See Section 12 for the calculations.

9.2 Daily continuing calibration verification is performed to ensure the initial calibration is still valid. The daily continuing calibration verification consists of the midlevel calibration standard and the instrument blank standard which consists of clean solvent spiked with the surrogate.

9.2.1 A midlevel continuing calibration verification standard is analyzed and the calculated value is compared against the average RF of the initial calibration or the curve (Section 9.1). The calibration is acceptable if the calculated value of the analytes are within +/- 15% of the expected value.

9.2.2 If the midlevel continuing calibration verification standard does not meet the acceptance criteria, then it is reanalyzed using a freshly prepared calibration standard. If it fails two consecutive times, for any reason, a new calibration curve is run.

9.3 An instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean. Blank criteria are defined in Section 10.2.

9.4 When analyzing samples, any extract that contains a target compound that exceeds the highest level calibration standard concentration must be diluted to within the calibration range.

9.5 The midlevel continuing calibration standard is run after every 10 or fewer samples. All compounds in the standard must yield values (+/-) 15% of their expected value to verify the continued working conditions of the instrument and acceptable performance for sample analysis.

9.5.1 If the midlevel continuing calibration standard fails to meet the above criteria, then it is reanalyzed. If it fails a second time, for any reason, a new calibration curve is to be run and all samples associated with the failing calibration verification standard must be reanalyzed.

10.0 QUALITY CONTROL

10.1 Method Detection Limit (MDL) studies are performed in accordance with the SOP for Conducting MDL Studies, IEA DOC #QAS02000.NET to determine the MDL for each target compound.

10.2 A one-time precision and accuracy determination for each analyst must be completed.

10.3 Refer to Table 1 for Practical Quantitation Limits (PQL's) for all compounds.

10.4 Daily Calibration Verification

10.4.1 A calibration (midlevel) standard and instrument blank are initially run daily at the start of the analysis run. The calibration standard must be within +/- 15% of expected value; and the blank must contain no peaks > PQL for all compounds. If the instrument blank does not meet criteria the autosampler, syringes and solvents must be checked for possible sources of interference and the system demonstrated to be clean before analysis of any samples can proceed.

10.5 Laboratory Control Sample

10.5.1 A Laboratory Control Sample (LCS) is analyzed with every batch of 20 samples or less. Recovery criteria are listed in Table 2. If these criteria are not met, then all samples in the batch are re-extracted and reanalyzed for the affected components.

10.6 Matrix Spike/Matrix Spike Duplicate

10.6.1 A Matrix spike (MS) and matrix spike duplicates (MSD) is analyzed with every batch of 20 samples or less. Recovery criteria are listed on Table 3.

10.6.2 If these criteria are not met, but the laboratory control sample (LCS) has acceptable recovery criteria, then the MS/MSD are documented as having matrix interferences. If the LCS fails the criteria listed in Table 2, then all samples in that batch are reextracted and reanalyzed.

10.6.3 The MS/MSD recoveries are evaluated for precision using the relative percent difference calculation found in Section 12.6. RPD limits are defined as +/- 35%. Results that are not within this limit should be evaluated but do not require corrective action.

10.7 Continuing Calibration Verification

10.7.1 A midlevel continuing calibration standard is analyzed every 10 samples and must have all components within +/- 15% of the expected value. If this criteria is not met, the continuing calibration standard must be prepared and reanalyzed within acceptable performance and all preceding samples associated with the failing continuing calibration standard must be reanalyzed.

10.8 Method Blank

10.8.1 A method blank must be analyzed with each extracted batch of samples. This should have no target compounds above the stated PQL's.

10.8.2 The method blank must be analyzed on each instrument where samples from the batch are analyzed. If the method blank contains target compounds above PQL's, it should be reanalyzed. If the contamination is confirmed with reanalysis, the extraction batch associated with the method blank is to be re-extracted and reanalyzed.

10.9 Surrogate Recovery

10.9.1 When surrogate recovery from a sample or method blank is <30% or >140%, check (1) calculations to locate possible errors, (2) fortifying solutions for degradation, (3) contamination, and (4) instrument performance. If these steps do not reveal the cause of the problem, reanalyze the extract.

10.9.2 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the recovery criterion, the sample should be re-extracted and re-analyzed to determine if sample matrix caused the surrogate failure.

10.9.3 Method blanks must have acceptable surrogate recovery. If surrogate recovery fails for a method blank after a confirming reanalysis, the blank and the extract batch should be reextracted and reanalyzed.

10.10 Retention Time Windows

10.10.1 Retention time windows are determined by making three injections of all standard mixtures throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

10.10.2 Calculate the standard deviation of the three retention times for each component. Plus or minus three times the standard deviation of the retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily on the interpretation of the chromatograms.

10.10.3 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

10.10.4 The laboratory must calculate retention time windows for each standard on each GC and whenever a new GC column is installed. The data must be retained by the laboratory and a copy provided to the QA department.

10.11 All positive results of analysis for sample extracts must be confirmed qualitatively by a second GC column or GC/MS. Results that do not confirm on a secondary column analysis are not reported.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 The following gas chromatographs are currently being used for Herbicide analysis under this SOP.

11.1.1 HP5890 Series II GC with dual EC's and 7673A Twin Tower Autosampler with columns and conditions as follows:

11.1.1.1

Front Injection port:	30 meter DB-1701 (See Section 7.1)
Column flow:	4.0 mL/min He
Detector Make-up Flow	55 mL/min P-5
Injector Temperature	Tracks oven temperature +3 deg. C
Detector Temperature	325 deg. C
Temperature Programming:	
Initial Temperature:	150 deg. C Hold 3.0 min
Initial Ramp:	4 deg/min to 220 deg. C, Hold 0.0 min.
Final Ramp and Temp.:	6 deg./min. to 270 deg. C, Hold 18 minutes

11.1.1.2	Back Injection Port	RTX-35 (See Section 7.1)
	Column Flow	4.0 mL/min. He
	Detector Make-up Flow:	55 mL/min. P-5
	Injector Temperature:	Tracks oven temperature +3 deg. C
	Detector Temperature:	325 deg. C
	Temperature Programming:	
	Initial Temperature:	150 deg. C, Hold 3.0 min.
	Initial Ramp:	4 deg./min. to 220 deg. C, Hold 0.0 min.
	Final Ramp and Temp:	6 deg./min. to 270 deg. C, Hold 18 min.

11.1.2 Tracor 540 with a single ECD and a 30 meter RTX-35 (See Section 7.1) with the following conditions:

Column Flow:	5.0 mL/min. He
Detector Make-up Flow	85 mL/min. P-5
Injector Temperature:	225 deg. C
Detector Temperature:	250 deg. C
Temperature Programming:	
Initial Temperature:	180 deg. C, Hold 10 min.
Initial Ramp:	10 deg./min.
Final Temp:	220 deg. C, Hold 5.0 minutes.

11.2 Sample Extraction: See sample preparation SOP for Herbicides in water by Method 8150, IEA Doc# SPS01001.NC.

11.3 After successful calibration verification, the herbicide extracts are set up on the autosampler to run on the gas chromatograph in the following sequence:

Herbicide Mid-level Standard
Instrument Blank
Herbicide Extraction Blank
Samples (up to 10)
Herbicide Mid-level Standard
Samples (up to 10)
LCS*
MS*
MSD*

*These QA samples may also be placed in other locations in the sequence.

11.4 All analyses are documented through the printed sequence log for the data file in use. The sequence log printouts for the instrument/data file in use is bound monthly with a sequence log cover page for each sequence verfied by the analyst (Attachment II). All sequence files for the month are verified with a printout of the list of files for each instrument also bound monthly.

11.5 Data System:

HP1000 Analytical Data System with A-400 CPU, running RTE-A Operating System and HP3350A Laboratory Automation System Software for data collection and reduction.

12.0 CALCULATIONS

12.1 Quantitation using average RF's: the RF for each compound is calculated as

$$\frac{\text{Area of peak}}{\text{Mass injected (ng)}} = \text{RF} \qquad \text{Avg RF} = \frac{\text{RF1}+\text{RF2}+\text{RF3}+\text{RF4}+\text{RF5}}{5}$$

12.1.1 Determination of sample concentration (waters)

$$\frac{\text{Area of peak}}{\text{Avg RF}} \times \frac{\text{Final extract vol.}}{\text{Initial sample vol.}} \times \text{Dilution Factor (DF)} = \text{sample conc. ug/L}$$

12.1.2 % Relative Standard Deviation (RSD):

SD / Avg RF x 100%

Where,

SD = Standard Deviation of average Rfs
Avg RF = Mean of 5 standard RFs

12.2 Quantitation using calibration curves: The calculation of target analytes concentration is done by the HP3350 software against the calibration curve using the following external standard calculation:

$$\text{Amount of y} = \frac{\text{(Corrected Response)y}}{\text{Vol. Ratio}} \times \frac{\text{Dilution factor}}{100}$$

where:

(Corrected Response)y = the Measured Response (area or height) of peak y after correction by the calibration curve.

Vol. Ratio = is the ratio of the volume of sample injected in this run to the volume of calibration mix injected in the calibration run. The default value is 1, meaning that the volumes are the same.

Dilution factor = the percent dilution factor, Default = 100. Any change in extract dilution is entered here.

12.3 Surrogate, LCS, Continuing Calibration Standard recoveries:

$$\%\text{Recovery} = \frac{\text{Amount recovered}}{\text{Amount Added}} \times 100\%$$

12.4 MS/MSD Recovery:

$$\%\ \text{Recovery} = \frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Spike Amount}} \times 100\%$$

12.5 % Difference (%D):

$$\%\ \text{Difference} = \frac{|\ \text{Calculated conc. - expected conc.}\ |}{\text{Expected conc.}} \times 100\%$$

12.6 Relative Percent Difference (RPD):

$$\text{RPD} = \frac{|\text{MS \%Recov - MSD \%Recov}|}{(\text{MS \%Recov +MSD \% Recov})/2} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 The 5-point initial calibration curve (Table 4) is considered acceptable if the %RSD is <20% or the calibration curve is evaluated.

13.2 Verification of the initial calibration curve with the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is within +/- 15% D of the expected value.

13.3 The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.5 Matrix Spike and Matrix Spike Duplicates are extracted and analyzed with every batch of up to 20 samples. Method acceptance limits are listed in Table 3.

13.6 Laboratory Control Samples are extracted and analyzed with every batch of up to 20 samples. Method acceptance limits are listed in Table 2.

13.7 Continuing Calibration (midlevel) Standards must be analyzed after every set of 10 samples or less. All analytes in the standard must yield values within (+/-) 15% of their expected value verses the calibration curve.

13.8 Surrogate recoveries must meet the 30-140% recovery limits. Should these limits not be met, the sample(s) must be reanalyzed. If, after reanalysis, the surrogate recovery is not within acceptance limits, the sample(s) must be re-extracted and reanalyzed.

14.0 REPORTING OF RESULTS

14.1

Units of measure:	mg/L
Significant figures:	2
Below Quantitation Limit:	BQL, no estimated (J) values are provided unless specifically requested by the client in advance.

14.2 A Level I report of results provides information on the following items to the client for samples and method blanks:

IEA ID for sample.
Client ID for sample.
Date of extraction.
Date of analysis.
Surname of analyst.
List of target compounds.
List of quantitation limits.
List of concentration data for each compound.
A comments section to describe various events during analysis (e.g., dilution factors, matrix interferences, etc.).
Copy of Sampling COCs.

14.3 A Level II report includes all data in Section 14.2 plus QC summary reports for surrogate and MS/MSD recovery.

14.4 A Level III report includes all data in Section 14.3 plus all raw data (i.e., LCS, calibrations), internal laboratory COCs and a project narrative.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for Extracting Herbicide Water Samples by Method 8150, IEA Doc# SPS01001.NC.

15.3 SOP for Solvent/Chemical Reagent Approval, IEA Doc# QAS00401.NET.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 8150.

16.2 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 8000.

17.0 SUBSTANTIVE REVISIONS

17.1 Original issue. (04/30/97)

TABLE 1
Practical Quantitation Limits (PQL)

Analyte	PQL, mg/L	Results, mg/L (2 sig.fig.)
Dinoseb	0.0020	BQL unless above PQL
2,4 - D	0.0020	BQL unless above PQL
Silvex	0.00040	BQL unless above PQL
Pentachlorophenol	0.0010	BQL unless above PQL
2,4,5 - T	0.0010	BQL unless above PQL

TABLE 2
Laboratory Control Sample Recovery Limits

Analyte	Spike Added, mg/L	Recovery Limits
2,4 - D	0.0040	70 - 120%
Silvex	0.00080	64 - 120%
Pentachlorophenol	0.0020	25 - 175%
2,4,5 - T	0.0020	28 - 110%

TABLE 3
Matrix Spike/Matrix Spike Duplicate Recovery Limits

Analyte	Spike Added, mg/L	Recovery Limits	RPD Limits
2,4 - D	0.0040	70 - 120%	35%
Silvex	0.00080	64 - 120%	35%
Pentachlorophenol	0.0020	25 - 175%	35%
2,4,5 - T	0.0020	28 - 110%	35%

TABLE 4
Calibration Concentrations, ug/mL

Analyte	LEVEL I	LEVEL II	LEVEL III	LEVEL IV	LEVEL V
2,4-D	0.20	0.40	0.50	1.0	2.0
Silvex	0.020	0.040	0.050	0.10	0.20
2,4,5-T	0.020	0.040	0.050	0.10	0.20
Pentachlorophenol	0.020	0.040	0.050	0.10	0.20
DCAA	0.10	0.20	0.25	0.50	1.0

ATTACHMENT I

Stock Standards Receiving Log
GC/GCMS Laboratory

Date Received	Analyst	Standard Name	Stock/Standard Code Lot #	Vendor	Expiration Date	Comments

Secondary Review By:________________ Date:__________

IEA, Inc. Form# QAL01200.NC

IEA Logbook# QA*

ATTACHMENT II

STANDARDS LOGBOOK

Standard Name: ______________
Location: ______________

Lot Code #: ______________
Solvent Used: ______________
Volume of Solvent Used: ______________

Prepared By: ______________
Date Prepared: ______________
Expiration Date: ______________

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc	Amt Added	Final Vol
1.						
2.						
3.						
4.						
5.						

Calculations:

Analyte	Final Analyte Concentration	Analyte	Final Analyte Concentration

Comments: ______________

Secondary Review By: ______________ Date: ______________

ATTACHMENT III

GC SEMIVOLATILE RUN SEQUENCE LOG

COVER PAGE

Instrument ID:__________________

Sequence Filename: _______________________

Analyst: _______________________

Analysis Start Date: _______________________

Method: _______________________

Sequence Verification By/Date:______________________________

22. IEA SOP FOR METALS DIGESTION METHOD 3005

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director ______________

Quality Assurance Manager ______________

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of surface water and ground water samples. The procedure is used to determine total recoverable metals or dissolved metals.

2.2 Samples prepared by this method may be analyzed by FLAA or ICP for the following:

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

2.3 The test codes for this document are MEPT51, METP52, and METP53.

3.0 SUMMARY OF METHOD

3.1 A mixture of HNO_3, HCl and material to be analyzed is refluxed in a 250 mL beaker. After the sample has been reduced to 15-20 mL the sample is brought to volume with DI water.

3.2 This method is based on EPA method 3005.

3.3 This method does not deviate from method 3005.

4.0 INTERFERENCES

4.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

4.2 Contamination can be a problem in sample preparation. Trace grade acids are used to reduce this problem. These chemicals are described in section 8.0. In addition, a clean work area is very important.

5.0 SAFETY

5.1 This method uses concentrated acids. These chemicals can cause severe burns and eye damage. Always wear gloves when handling samples or reagents. Wear face shield, lab coat or apron to help protect against spills and splashes. Wear respirator to protect against acid fumes and fumes from samples. Keep hood closed while samples are digesting.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Samples should be received at pH <2.

6.2 Final digestates are to be placed in new 125 mL plastic bottles. Sample bottles are not to be reused.

6.3 Samples are stable when digestion is complete and need no preservation.

6.4 Holding time is 180 days.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Ventilation Hood

7.1.2 Hotplates

7.1.3 250 mL beakers

7.1.4 Watchglasses

7.1.5 Glass Rods

7.1.6 Graduated Cylinders

7.1.7 100 mL volumetrics

7.1.8 pH paper

7.1.9 Plastic bottles

7.1.10 Whatman number 42 filter paper

7.1.11 Funnels

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Barnstead deionized water, 16.6 megohm or higher

8.2 Concentrated Nitric Acid, Trace Grade

8.3 Concentrated Hydrochloric acid, Trace Grade

8.4 For preparation codes METP52 and METP53, no sample spikes or LCSW are performed as this is a dissolved undigested prep.

8.5 For preparation code METP51 the sample spike receives 1.00 mL of IEA-SPK-2 and 1.00 mL of IEA-SPK-1 for every 100 mL digested. The LCSW receives 1.00 mL of IEA-ICV-3 and 1.00 mL of IEA-ICV-1 for every 100 mL digested. Samples requiring B, Mo, Si, Sn, or Ti also receive 1.00 mL of IEA-ICV-2 for every 100 mL of LCSW digested.

9.0 CALIBRATION

9.1 No calibration is required for this method.

10.0 QUALITY CONTROL

10.1 Preparation blanks are performed to check for contamination. If a preparation blank is above the reporting limit and the samples are not a least ten times the preparation blank value then the samples in the batch are reprepped. The exception to this are if none of the samples are above the reporting limit then the batch is not reprepped.

10.2 A lab control sample is prepared to check that no analyte is lost or gained during the batch preparation. If the lab control sample does not meet the $\pm$ 20 % of the true value or the internally established control limits (whichever is more stringent), then the entire batch is reprepped. The exceptions to this are silver and antimony.

10.3 A duplicate sample is prepared and the RPD is calculated from the original sample. If the RPD is greater than 20 % and the sample is greater than five times the reporting limit the samples in that batch are flagged. If the sample is less than five times the reporting limit then the control limit is $\pm$ the reporting limit.

10.4 A sample spike is also performed each batch. This must agree within $\pm$ 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.5 Control Charts are used to track the results of the lab control sample over time. Trends are noted to evaluate possible future problems such as analytes falling out of solution and the problem solved (i.e., replacing the solution or standard).

10.6 A sample spike duplicate is also performed each ICAP batch. This must agree within $\pm$ 20 % RPD with the original sample spike.

10.7 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every 10 samples
Sample Spike Duplicate	Every Batch not to exceed 20 samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Using glass rods, take pH of sample and preserve if necessary.

11.2 Using a graduated cylinder, transfer a 100 mL representative aliquot of the well mixed sample to a 250 mL beaker and add 2 mL of the concentrated HNO_3 and 5 mL of concentrated HCl.

11.3 Place the beaker on a hotplate and cautiously evaporate to a 15-20 mL volume making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to dry.

11.4 Cool the beaker. Wash down the beaker walls and when necessary, filter sample to remove insoluble material. Before bringing the sample to volume add 3 mL of HNO_3 to the sample. Bring to volume with DI water to 100 mL in a volumetric flask. Place sample in a new 125 mL plastic bottle. The sample is now ready for analysis.

12.0 CALCULATIONS

12.1 None.

13.0 ACCEPTANCE OF DATA

13.1 Acceptance of the data is described in section 10.0

14.0 REPORTING OF RESULTS

14.1 Each log book page should have a date, analyst's name and batch number.

14.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in the log book.

14.3 Record pH, matrix, initial volume, and final volume for each sample.

14.4 Record lot number of all solutions and reagents added.

14.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None

16.0 REFERENCES

16.1 Method 3005A, Acid Digestion of Waters for Total Recoverable Metals for Analysis by FLAA or ICP Spectroscopy, SW-846 Test Methods for Evaluating Solid Waste, July 1992 Revision.

16.2 Employee Chemical Safety Handbook, Industrial and Environmental Analysts, Inc., 1990.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and sample spike and LCSW spiking levels were updated in this revision.

17.2 Addendum to Section 10.2: Control limit criteria added. Sections 10.6 and 10.7 were revised to include the MSD for ICAP analysis. Section 10.7 amended: Duplicate changed from every 20 to every 10 per NC. Section 11.4 was modified to include the addition of 3 mL HNO_3 for matrix match (sample and standard) purposes.

23. IEA SOP FOR METALS DIGESTION METHOD 3010

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director ______________________

Quality Assurance Manager ______________________

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of aqueous samples, EPTOX, Regulatory and Non-Regulatory TCLP extractions, and wastes that contain suspended solids for analysis by AA or ICP. The procedure is used to determine total metals.

2.2 Samples prepared by this method may be analyzed by FLAA or ICP for the following:

Aluminum	Lead
Arsenic	Magnesium
Barium	Manganese
Beryllium	Molybdenum
Cadmium	Nickel
Calcium	Potassium
Chromium	Selenium
Cobalt	Sodium
Copper	Thallium
Iron	Vanadium
	Zinc

2.3 The test codes for this document are METP15, METP50, and METP45.

3.0 SUMMARY OF METHOD

3.1 A mixture of HNO_3 and material to be analyzed is refluxed in a 250 mL beaker. This step is repeated with additional portions of HNO_3 until digestate is light in color or until its color has stabilized. After digestate has been brought to a low volume, it is refluxed with HCl and brought up to volume for the ICP. If the sample should go to dryness, it must be discarded and sample reprepared.

3.2 This method is based on EPA method 3010.

3.3 This method does not deviate from method 3010.

4.0 INTERFERENCES

4.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

4.2 Contamination can be a problem in sample preparation and trace grade acids are used to reduce these problems. These chemicals are described in section 8.0. In addition, a clean work area is very important.

5.0 SAFETY

5.1 This method uses concentrated acids. These chemicals can cause severe burns and eye damage. Always wear gloves when handling samples or reagents. Wear face shield, lab coat or apron to help protect against spills and slashes. Wear respirator to protect against acid fumes and fumes from samples. Keep hood closed while samples are digesting.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Final digestates are to be placed in new 125 mL plastic bottles. Sample bottles are not to be reused.

6.2 Samples are stable when digestion is complete and need no preservation.

6.3 Holding time is 180 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Ventilation Hood

7.1.2 Hotplates

7.1.3 250 mL beakers

7.1.4 Watchglasses

7.1.5 Glass Rods

7.1.6 Graduated Cylinders

7.1.7 100 mL volumetrics

7.1.8 pH paper

7.1.9 Plastic bottles

7.1.10 Whatman number 42 filter paper

7.1.11 Funnels

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Barnstead deionized water, 16.6 megohm or higher

8.2 Concentrated Nitric Acid, Trace Grade

8.3 1:1 Hydrochloric acid, mix 8.1 with trace grade hydrochloric acid

8.4 For preparation codes METP52 and METP53 no sample spikes or LCSW are performed as this is a dissolved undigested prep.

8.5 For preparation code METP51 the sample spike receives 1.00 mL of IEA-SPK-2 and 1.00 mL of IEA-SPK-1 for every 100 mL digested. The LCSW receives 1.00 mL of IEA-ICV-3 and 1.00 mL of IEA-ICV-1 for every 100 mL digested. Samples requiring B, Mo, Si, Sn, or Ti also receive 1.00 mL of IEA-ICV-2 for every 100 mL of LCSW digested.

8.6 For preparation code METP15 samples spikles are spiked in the leachate lab. LCSW samples receive 2.0 mL of TCLP SPIKE/LCSW STATE LEVELS for every 100 mL digested.

8.7 For preparation code METP45 sample spikes are spiked in the leachate lab. LCSW samples receive 1.0 mL of FEDERAL SPIKE A for every 100 mL of LCSW digested.

9.0 CALIBRATION

9.1 No calibration is required for this method.

10.0 QUALITY CONTROL

10.1 Preparation blanks are performed to check for contamination. If a preparation blank is above the reporting limit and the samples are not a least ten times the preparation blank value then the samples in the batch are reprepped. The exception to this are if none of the samples are above the reporting limit then the batch is not reprepped.

10.2 A lab control sample is prepared to check that no analyte is lost or gained during the batch preparation. If the lab control sample does not meet the ± 20 % of the true value then the entire batch is reprepped. The exceptions to this are silver and antimony.

10.3 A duplicate sample is prepared and the RPD is calculated from the original sample. If the RPD is greater than 20 % and the sample is greater than five times the reporting limit the samples in that batch are flagged. If the sample is less than five times the reporting limit then the control limit is ± the reporting limit.

10.4 A sample spike is also performed for each batch. This must agree within ± 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.5 Control Charts are used to track the results of the lab control sample over time. Trends are noted to evaluate possible future problems such as analytes falling out of solutions and the problem solved, i.e., replacing the solution or standard.

10.6 A sample spike duplicate is also performed for each ICAP batch. This must agree within ± 20 % RPD with the sample spike.

10.7 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every 10 samples
Sample Spike Duplicate	Every Batch not to exceed 20 samples

10.7.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled per day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Using glass rods, take pH of sample and preserve if necessary.

11.2 Using a graduated cylinder, transfer a 100 mL representative aliquot of the well mixed sample to a 250 mL beaker and add 3 mL of the concentrated HNO_3.

11.3 Place the beaker on a hotplate and cautiously evaporate to a low volume (5 mL - 25 mL) making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to dry. Cool the beaker and add another 3 mL portion of concentrated HNO_3. Increase the temperature of the hotplate so that a gentle reflux action occurs.

11.4 Continue heating, adding additional acid if necessary, (no more than 10 mL) until the digestion is complete, (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Evaporate to a low volume not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add 10 mL 1:1 HCl and warm the beaker for an additional 15 minutes to dissolve and precipitate or residue resulting from evaporation.

11.5 Wash down the beaker walls with DI water and when necessary, filter sample to remove insoluble material. Bring to 100 mL final volume with DI water. Place sample in a new 125 mL plastic bottle. The sample is now ready for analysis.

12.0 CALCULATIONS

12.1 None.

13.0 ACCEPTANCE OF DATA

13.1 Acceptance of the data is described in section 10.0

14.0 REPORTING OF RESULTS

14.1 Each log book page should have a date, analyst's name and batch number.

14.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in the log book.

14.3 Record pH, matrix, initial volume, and final volume for each sample.

14.4 Record lot number of all solutions and reagents added.

14.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None

16.0 REFERENCES

16.1 Method 3010, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, September 1986.

16.2 Employee Chemical Safety Handbook, Industrial and Environmental Analysts, Inc., 1990.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and sample spike and LCSW spiking levels were updated in this revision.

17.2 Addendum to Section 6.3 to clarify holding time. Section 8.1 ammended from 18.0 to 16.6 mega-ohm d.i. reagent water. Addendum to Section 10.6 to include definitions.

17.3 Revision to Section 2.3: Test codes METP17, METP47, METP51, METP52, METP53 omitted. Test code METP50 added. Sections 10.6 and 10.7 were updated to include the MSD for ICAP analysis. Section 10.7 revised: Duplicate changed from 20 samples to every 10 per NC.

24. IEA SOP FOR METALS DIGESTION METHOD 3015

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to describe the digestion procedure used to prepare aqueous samples, TCLP extracts and wastes that contain suspended solids for analysis by FLAA, ICAP, GFAAS or ICP/MS. Samples are digested using a microwave oven with temperature and pressure feedback.

2.2 This procedure is applicable to the following analytes:

Aluminum	Antimony	Arsenic	Barium
Beryllium	Cadmium	Calcium	Chromium
Cobalt	Copper	Iron	Lead
Magnesium	Manganese	Molybdenum	Nickel
Potassium	Selenium	Silver	Sodium
Thallium	Vanadium	Zinc	

2.3 The Test Codes applicable to this SOP are METP91, METP92 and METP93.

3.0 SUMMARY OF METHOD

3.1 A 45 mL aliquot of sample is digested using 5 mL of concentrated nitric acid in a Teflon microwave vessel. The sample is then cooled and placed in a plastic bottle.

3.2 This SOP is based on SW-846 Method 3015.

3.3 This SOP does not deviate from the above listed method.

4.0 INTERFERENCES

4.1 Samples with high organic matter will result in elevated vessel pressure. Venting will result in the loss of sample and/or analyte. The sample must not be vented. A smaller volume of sample diluted to 45 mL may be required to prevent this interference, resulting in elevated detection limits.

5.0 SAFETY

5.1 This procedure uses concentrated nitric acid. Care should be used when handling this acid and all chemicals. Strong acids cause severe burns to the skin and other soft tissues. Lab coats, gloves and safety glasses should be worn when using acids. Nitric acid fumes cause irritation of the eyes and nose and should be used in a fume hood.

5.2 Microwave ovens used in this method produce microwave radiation and should never be operated with the door open.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Samples should be collected in glass or plastic bottles.

6.2 Samples should be preserved with nitric acid.

6.3 Holding times for samples is 180 days from collection.

7.0 APPARATUS AND MATERIALS

7.1 1000 Watt Microwave Oven with computer control.

7.2 Teflon vessels with pressure relief caps, rupture disks and liners.

7.3 Sensor vessel and associated rupture disk.

7.4 Vessel Rack.

7.6 Portable hotplate.

7.7 Electronic Balance capable of weighing up to 900 grams to two decimal places.

7.8 50 mL graduated cylinder.

7.9 Plastic digestate storage containers.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Trace Metals Grade Concentrated Nitric Acid.

8.2 Ice.

8.3 DI water 16.6 megohm-cm or higher.

8.4 10% Aqua regia, Add 7.5 mL of HCl and 2.5 mL of HNO3 to a beaker. Dilute to 100 mL with DI water.

8.5 Matrix Spiking Solution, prepared as specified in the appropriate and corresponding referenced analytical SOP. (See Section 15.0.)

8.6 Laboratory Control Sample solution, prepared as specified in the appropriate and corresponding analytical SOP. (See Section 15.0.)

8.7 Reagent receipt is documented in the Chemical Receipt Logbook (Attachment I).

9.0 CALIBRATION

9.1 The temperature calibration is performed as follows, in accordance with manufacturer's instructions:

9.1.1 From the Main Menu select "Calibrate".

9.1.2 Select F2 for temperature calibration. Select the control vessel to calibrate (A for the first vessel, B for the second vessel, etc.) and press enter. The computer will prompt you to "Place the probe in 0 °C water and allow the temperature to stabilize for 10 seconds".

9.1.3 Place the probe in a Teflon beaker full of crushed ice and DI water. Ensure that the water is well mixed and full of ice. Wait for the reading to stabilize. Ten seconds after the reading stabilizes press enter. The zero point is now set.

9.1.4 The computer will now prompt you to "Place the probe in 100 °C water and allow the temperature to stabilize for 10 seconds". Place a hotplate near the microwave, but not inside the microwave. Bring a beaker of DI water to a vigorous boil. Place the probe in the beaker and wait for the temperature to stabilize. Ten seconds after the reading stabilizes press enter. The temperature calibration is now complete.

9.2 The pressure calibration is performed as follows, in accordance with the manufacturer's instructions:

9.2.1 From the Main Menu select "Run Method". Press F1 and select "CAL170". Make note of the data file used by the method. Fill the control vessel with 30 mL DI water and place rupture disk in place. Seal the vessel. Run the method by pressing F3. You will be prompted for the control vessel (A, B, etc.). When the vessel has been selected, the run will start. When the run is complete, return to the main menu.

9.2.2 From the Main Menu select "Calibrate".

9.2.3 Select F3 for pressure calibration. A message with detailed information regarding calibration will appear. Press enter. Select the control vessel and press enter. Select the data file from CAL170.MTH and press enter. If no errors are present the screen will display "Calibration Complete".

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are calculated in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET from samples prepared according to this SOP and analyzed by an acceptable corresponding method SOP.

10.2 Duplicates are prepared one per ten samples or per batch, whichever is more frequent.

10.3 Matrix Spikes are prepared one per twenty samples or per batch, whichever is more frequent. Spike 0.50 mL of spiking solutions into the matrix spike sample aliquot.

10.4 Matrix Spike Duplicates are prepared every twenty samples or per batch, whichever is more frequent. Spike 0.50 mL of spiking solutions into the matrix spike duplicate sample aliquot.

10.5 Preparation Blanks are prepared one per twenty or per batch, whichever is more frequent.

10.6 Laboratory Control samples are prepared one per twenty or per batch, whichever is more frequent. Spike 0.450 mL of spiking solutions into the LCSW.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Prepare the digestion vessel by heating 10% Aqua Regia in a beaker on a hotplate to 80° C. Rinse the Teflon liners with the hot Aqua Regia. Rinse the Teflon liners three times with DI water. **Never use a brush or wipe the liners with a paper towel as this may scratch the liner surface.** Liners that have scarring or cracks should be inspected by the supervisor and replaced if needed. Clean a 50 mL graduated cylinders in the same manner.

11.2 Weigh the complete digestion vessel, including the rupture disk to 0.01 g. Record the weight of the vessel and the vessel number in the Metals Digestion Logbook (Attachment II).

11.3 Shake the sample and measure exactly 45 mL of sample in a 50 mL graduated cylinder. Pour the sample into the digestion vessel and add 5 mL of concentrated nitric acid using the repipetor to each sample. Allow the samples to vent gases for five minutes or until the sample stops effervescing. Place the rupture disk in place and cap the vessel. Tighten the cap until resistance is felt and then tighten slighlty more (approximately an 1/8 of a turn). Select one representative sample for use in the control vessel. (Do not use the LCSW or the PBW.) Ensure that the outside of the vessels and the microwave cavity are dry.

11.4 Weigh the complete digestion vessel, including the rupture disk and sample to 0.01 g. Record the weight of the vessel and the vessel number in the Metals Digestion Logbook.

11.5 Place the vessels in the tray with the vent tube ends in the vent container. Plug the control vessel into the socket in the microwave cavity. The plug has a has a notch that, when introduced to the socket at the twelve o'clock position, will be in proper alignment with its counterpart. Close the door and ensure that the table rotates freely.

11.6 From the Main Menu select "Run Method". Press F1 to select the method. A list of methods will appear. Highlight and select "EPA3015". Press F2 to select the data file. Highlight "New" and type in the eight character batch ID (MMDDYYNN i.e., 01219501). The computer will prompt "Do you want to enter a notebook entry? (y/n)". Press "Y" and then enter. When the Notebook screen appears, enter the appropriate data. When complete press F10 to return to the Run Control screen. Ensure that the interlock button on the left side is out. You may stop the magnitron at any time by pressing the interlock button in. To start the run press F3. Select the control vessel in capital letters (A or B, see the control top). Press Enter and the run will begin. During the run press F4 to toggle between the graph and the run screen. Pressing F5 will toggle between the method screen and the run screen. F3 will stop the run. You will be prompted "Are you sure you want to STOP RUN (y/n):". Press y or n as needed. Press F10 to exit the screen when the run is complete.

11.7 Remove the vessels from the microwave by removing the entire rack after disconnecting the control vessel cable. Allow the samples to cool for five minutes in the rack. The samples may be cooled in a water bath, but ensure that the water is not above the threads. After the samples reach room temperature wipe the outside dry and weigh each vessel to 0.01 grams. Record the weight in the Metals Digestion Logbook. If more than 10% of the sample weight has been lost, the sample has vented and must be discarded.

11.8 Open each vessel one at a time and drain any condensation from the rupture disk to the liner. Remove the liner and transfer the sample to a new plastic bottle. Ensure that all condensation is transferred by turning the liner as it is poured. Do not rinse the liner to transfer the sample. Record the final volume in the Microwave Digestion Logbook as 50 mL.

11.9 Wash the vessels and liners with DI water. **DO NOT USE brushes to clean the liners.** Discard the rupture disks.

12.0 CALCULATIONS

12.1 Sample results are calculated as follows.

Preparation Factor = Final Volume/Initial Volume

Preparation Factor = 50 mL/45 mL

12.2 To calculate the loss of sample due to venting use the following formula.

$$\%Loss = \frac{(WB - WV) - (WA - WV)}{(WB - WV)} \times 100\%$$

WB = Weight of Sample and Vessel before digestion
WA = Weight of Sample and Vessel after digestion
WV = Weight of Vessel

13.0 ACCEPTANCE OF DATA

13.1 Sample data is acceptable for digestion if 10% or less of the sample is lost due to venting.

14.0 REPORTING OF RESULTS

14.1 Results are reported using ACS. This procedure is described in the SOP for ACS Data Reporting, IEA Doc# MES02900.NC.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for ACS Data Reporting, IEA Doc# MES02900.NC.

15.3 SOP for Metals Analysis by ICPMS Method 6020, IEA Doc# MES03200.NC.

15.4 SOP for SW-846 Method 6010 with TJA 61E ICAP Operation, IEA Doc# MES00801.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 3015.

16.2 QWave 3000 Operation Manual, Questron Corporation.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 10.2 revised to reflect duplicate frequency every 10 samples, or per batch. Revised 03/13/96.

Attachment I

Metals Lab Chemical Receipt Log

DATE	LOT #	VENDOR	REFERENCE #	GRADE	QTY	VOLUME/MASS	ANALYST

Secondary Review:____________________ By:______________

Attachment II

Metals Digestion Log

IEA Sample #	EPA Sample #	pH	Microwave Digestion			Hotplate Digestion	Final Volume (mL)	Filter Data				Texture/Artifacts/Comments
			Vessel Wt (g)	Vessel + Sample Wt (g)	Wt After Digestion (g)	Sample Vol/Wt (mL/g)		Color		Clarity		
								Before	After	Before	After	

Batch #		Digestion Date		Matrix Spike #1 Lot #		LCS Lot #	
Method		HNO3 Lot #		MS #1 Spike Volume			
Dig. Analyst		HCL Lot #		Matrix Spike #2 Lot #			
Applicable SOP		H2O2 Lot #		MS #2 Spike Volume			
Matrix				Witness/Date			

Secondary Review By:________________ Date:__________

25. IEA SOP FOR NON-VOC AND INORGANIC TCLP PREPARATION

26. IEA SOP FOR HEXAVALENT CHROMIUM IN SOIL

27. IEA SOP FOR HEXAVALENT CHROMIUM IN WATER

28. IEA SOP FOR TOC IN SOIL METHOD 415.1

29. IEA SOP FOR TOC IN SEDIMENT

30. IEA SOP FOR TOTAL PHOSPHOROUS ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 Polyphosphates and organic phosphorus compounds are converted to orthophosphate by a sulfuric acid/persulfate digestion. Ammonium molybdate and antimony potassium tartrate then react in an acid medium with orthophosphate to form an antimony-phosphomolybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

2.2 This procedure is applicable to drinking, surface, saline, and ground waters; domestic and industrial wastes, both solid and liquid; and soils and sludges.

2.3 This procedure is applicable to Test Codes P_01 and P_02.

3.0 SUMMARY OF METHOD

3.1 An aliquot or weight of sample is digested with sulfuric acid and potassium persulfate. The resulting solution is filtered, if necessary, to remove solid particles, and mixed with a color reagent containing sulfuric acid, antimony potassium tartrate, ammonium molybdate, and ascorbic acid. After 10 minutes but no more than 30 minutes the absorbance is read on a spectrophotometer at 880 nm. The concentration of phosphorus is determined from comparison with a calibration curve.

3.2 This procedure is based on Standard Methods 18th Edition, 4500-P B and 4500-P E, and on EPA Method 365.2.

3.3 This procedure deviates from the above methods as follows:

3.3.1 100 mL of sample is digested instead of 50 mL.

3.3.2 The pH is not adjusted prior to digestion.

3.3.3 2 mL of 11N sulfuric acid and 1 g of potassium persulfate are used instead of 1 mL and 0.5 g, respectively.

3.3.4 The pH is adjusted after digestion.

3.3.5 A 50 mL aliquot is taken from the 100 mL digested solution for colorimetric testing.

3.3.6 Antimony potassium tartrate is stored in a dark bottle at 4 degrees C. Ammonium molybdate date is stored in a plastic bottle at 4 degrees C.

3.3.7 Phosphorus stock solution is 500 mg/L; intermediate standard is 50 mg/L; working standard is 2.5 mg/L.

3.3.8 This procedure has been modified to accommodate the analysis of soils/sludges.

4.0 INTERFERENCES

4.1 High iron concentrations can cause precipitation of and subsequent loss of phosphorus. Dilution of samples prior to digestion may be necessary to reduce iron interference.

4.2 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination.

4.3 Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

5.0 SAFETY

5.1 Concentrated sulfuric acid can cause severe burns. Goggles or a face shield, lab coat, and gloves are necessary when using this reagent. Gloves are recommended when using dilute solutions of sulfuric acid.

5.2 Potassium persulfate is a strong oxidizer; contact with combustible materials may cause fire. This reagent is harmful if inhaled or swallowed, and may cause skin irritation. Gloves are recommended when handling this reagent.

5.3 Sodium hydroxide can cause severe burns. Goggles or a face shield, lab coat, and gloves are recommended when preparing solutions of this reagent. Gloves are recommended when using dilute solutions of sodium hydroxide.

5.4 Gloves are recommended when handling antimony potassium tartrate, ammonium molybdate, and ascorbic acid; avoid inhalation and contact with skin.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Water samples are preserved with sulfuric acid to pH less than 2 with HCl at the time of collection. Samples are stored at 4 °C until analysis.

6.3 Soil samples are cooled to 4 °C for transport and storage until analysis.

6.4 The holding time for total phosphorus analysis is 28 days from sampling. If samples are delayed in transit or received after the 28 day holding time, the client is notified and a resampling schedule is arranged.

7.0 APPARATUS AND MATERIALS

7.1 Spectronic 1201 spectrophotometer; 1 cm square cuvet.

7.2 Top-loading balance, reading to 0.1 g.

7.3 250 mL beakers, funnels, 100 mL and 1 Liter volumetric flasks, and 125 mL Erlenmeyer flasks pre- rinsed with 50% hydrochloric acid and reagent water.

7.4 #40 filter paper.

7.5 Oxford pipetter, 5-10 mL.

7.6 Assorted beakers, graduated cylinders, transfer pipets, volumetric pipets, scoopulas.

7.7 pH Meter

7.8 Hot plate

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm-cm or higher, deionized.

8.1.2 Concentrated sulfuric acid (H_2SO_4), ACS grade.

8.1.2.1 11N H_2SO_4: Carefully add 310 mL concentrated H_2SO_4 to 500 mL reagent water in a 1-liter volumetric flask; swirl to mix; cool and dilute to volume.

8.1.2.2 5N H_2SO_4: Add 70 mL concentrated H_2SO_4 to 350 mL reagent water in a 500 mL volumetric flask; swirl to mix; cool and dilute to mark.

8.1.3 Potassium persulfate ($K_2S_2O_8$), ACS grade.

8.1.4 Ethanol, 95%, not denatured.

8.1.5 Sodium hydroxide (NaOH), ACS grade.

8.1.5.1 6N NaOH: Dissolve 120 g NaOH pellets in reagent water; cool and dilute to 500 mL.

8.1.6 Phenolphthalein indicator, ACS grade, purchased from a commercial vendor.

8.1.6.1 Dissolve 0.5 g phenolphthalein in 50 mL 95% ethanol and 50 mL reagent water.

8.1.7 Antimony potassium tartrate [$K(SbO)C_4H_4O_6$: ½ H_2O], ACS grade.

8.1.7.1 Antimony potassium tartrate: Dissolve 1.3715 g in reagent water; dilute to 500 mL. Store at 4 °C in a dark bottle.

8.1.8 Ammonium molybdate [$(NH_4)_6Mo_7O_{24}$: 4 H_20], ACS grade.

8.1.8.1 Ammonium molybdate: Dissolve 20 g in 500 mL reagent water. Store at 4 °C in a plastic bottle.

8.1.9 L-ascorbic acid, ACS grade

8.1.9.1 Ascorbic acid: Dissolve 1.76 g in 100 mL reagent water. Prepare fresh at time of analysis.

8.1.10 Combined color reagent: Mix, in order, per 100 mL of reagent:

50 mL 5N H_2SO_4 (Section 8.1.2.2)
5 mL antimony potassium tartrate (Section 8.1.7.1)
15 mL ammonium molybdate (Section 8.1.8.1)
30 mL ascorbic acid (Section 8.1.9.1)

8.1.11 Mix after addition of each reagent. Reagents should be at room temperature before mixing and must be mixed in the order given. Prepare fresh at time of analysis.

8.1.12 All reagent receipt and preparation is documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standards

8.2.1 Stock/Intermediate/Working Standards

8.2.1.1 Potassium dihydrogen phosphate (KH_2PO_4), dried at 105 °C, ACS grade.

8.2.1.1.1 Stock phosphorus solution, 500 mg/L: Dissolve 1.0985 g dried KH_2PO_4 in reagent water; dilute to 500 mL. Prepare annually.

8.2.1.1.2 Intermediate standard, 50 mg/L: Dilute 10.0 mL of Stock to 100 mL in reagent water. Prepare semi-annually.

8.2.1.1.3 Working standard, 2.5 mg/L: Dilute 50.0 mL of Intermediate standard to 1000 mL in reagent water. Prepare monthly.

8.2.2 Calibration Standards

8.2.2.1 Calibration standards are prepared by the dilution of Working Standard (Section 8.2.1.1.3) to 100 mL in reagent water according to the following table:

Volume of Working Standard (2.5 mg/L)	Calibration Standard Concentration, mg/L
0	0
0.8	0.020
5.0	0.125
15.0	0.375

25.0	0.625
40.0	1.000

The pH of each standard is adjusted with one drop each of phenolphthalein, 6N NaOH, and 11N H_2SO_4 prior to bringing to volume.

8.2.3 Initial and Continuing Calibration Verification Standards (ICV & CCV)

8.2.3.1 ICV/CCV Stock, 25 mg/L, 10 mL voluette, purchased from Hach, Cat.# 21092-10.

8.2.3.2 ICV/CCV Standard, 0.50 mg/L: Empty two 10 mL voluettes (Section 8.2.3.1) into a 1000 mL volumetric flask containing 900 mL reagent water and dilute to volume with reagent water.

8.2.4 Matrix Spike Standard

8.2.4.1 The intermediate standard (Section 8.2.1.1.2) is used to prepare a matrix spike standard at a concentration of 0.25 mg/L as in Section 10.3.

8.2.4.1.1 Pipet 500 uL of the intermediate standard (50 mg/L) into the sample prior to digestion.

8.2.5 All standard receipt and preparation is documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 Set the absorbance on the Spectronic 1201 at zero using the zero calibration standard (Section 8.2.2.1) and record the absorbance in the Spectrophotometric Analysis Logbook (Attachment III).

9.2 Analyze the calibration standards in succession from the lowest to the highest concentration (Section 8.2.2.1) and record the absorbances.

9.3 Correlation coefficients are calculated using linear regression.

9.4 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. concentration to be equal to or greater than 0.995.

9.5 The working calibration range of this method is 0.02 mg/L to 1.00 mg/L.

9.6 Calibrate the pH meter to be used for pH adjustment according to the SOP for pH, IEA Doc #CVS02400.NC.

9.7 Calibrate the balance daily, prior to use, according to the SOP for Calibration of Lab Balances, IEA Doc #QAS01002.NET.

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The Practical Quantitation Limits (PQLs) for this procedure is the MDL, currently 0.02 mg/L for waters and 20 mg/kg for soils.

10.3 Matrix spikes are prepared as in Section (8.2.4.1.1) and should be performed one per matrix per 10 samples or less being analyzed.

10.3.1 Matrix spike recoveries are determined as in Section 12.4 and should meet the criteria established in Section 13. Recoveries that do not meet this criteria may be attributable to the sample matrix effect.

10.4 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively). ICV/CCVs are prepared by processing 100 mL of ICV/CCV Standard, 0.50 mg/L (Section 8.2.3.2) as a sample.

10.4.1 Recovery calculations and criteria are found in Sections 12 and 13 and must be met.

10.5 Blanks, including the ICB, CCB and Preparation blank, are prepared by processing 100 mL of reagent water as a sample.

10.5.1 Failure to meet acceptance criteria as outlined in Section 13 will require reanalysis for samples associated with the ICB/CCB. Sample will require repreparation and reanalysis for failure of the Preparation blank.

10.6 Internal standards and surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per batch
Continuing Calibration Blank	Every 10 samples, per batch
Matrix Spike	Every 10 samples, per batch, per matrix
Sample Duplicate	Every 10 samples, per batch

10.7.1 DEFINITIONS:
Batch: Every 10 samples prepared, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrument Conditions

11.1.1 Turn on the Spectronic 1201 and allow the instrument to complete the Self-Check.

11.1.2 Set the wavelength to 880 nm and allow the instrument to warm up for at least 30 minutes.

11.1.3 Place the square cuvet holder in the chamber.

11.2 Sample Preparation

11.2.1 Water samples: Pour 100 mL of sample into a pre-rinsed 250 mL beaker. (A smaller aliquot diluted to 100 mL in reagent water may be used). Record the volume of sample used in the Spectrophotometric Analysis Logbook (Attachment III). Continue as in Section 11.2.3.

11.2.2 Soil samples: Measure 1.0 g of sample into a pre-rinsed 250 mL beaker and add 100 mL of reagent water. Record the weight of the sample used in the Spectrophotometric Analysis Logbook (Attachment III).

11.2.3 Add 2 mL 11N H_2SO_4 and 1 g potassium persulfate to the beaker and swirl to mix.

11.2.4 Place the beaker on a hot plate and boil gently until the volume is reduced to 10-15 mL. Remove the beaker from the hotplate and allow to cool.

11.2.5 Rinse the sides of the beaker with reagent water.

11.2.5.1 Place the pH electrode in the solution; additional reagent water may be needed to ensure the electrodes are fully submerged.

11.2.6 Adjust the pH of the solution in the beaker to between 6.8 and 7.2 using dilute NaOH or H_2SO_4, as necessary.

11.2.7 Remove the beaker after the pH adjustment. If sample has turned golden yellow and/or formed a floc during the pH adjustment, add 11N H_2SO_4 until color is dissipated and/or floc is redissolved.

11.2.8 Check the sample for turbidity or solid matter and filter, if necessary, through #40 paper into a pre-rinsed 100 mL volumetric flask. Rinse the beaker 3 times with reagent water, and filter the rinses. Rinse the filter 3 times with reagent water.

11.2.8.1 Remove the filter and rinse the funnel into the flask. Continue as in Section 11.2.11.

11.2.9 If filtration is not necessary, transfer the sample to a 100 mL volumetric flask.

11.2.10 Rinse the beaker 3 times with reagent water and add the rinses to the flask.

11.2.11 Dilute to volume and cap and shake well. Note any turbidity, color, or precipitate in the prepared sample. Do not refilter to remove turbidity or precipitate.

11.3 Analytical Procedure

11.3.1 Transfer 50 mL of the digested solution to a pre-rinsed 125 mL Erlenmeyer flask.

11.3.2 Add 8 mL of combined color reagent and swirl to mix.

11.3.3 After at least ten minutes, but no longer than 30 minutes, begin analysis of the samples by introducing the standards and sample(s) into the cuvets in succession (Section 9.2) and recording the absorbances of each in the analysis logbook.

11.3.4 The cuvet is rinsed with reagent water and the sample to be analyzed between each sample analysis.

12.0 CALCULATIONS

12.1 Water samples:

$$\text{mg/L P} = \text{mg/L from curve} \times \frac{100\text{ mL}}{\text{mL Sample}}$$

12.2 Soil samples:

$$\text{mg/Kg} = \text{mg/L from curve} \times \frac{100\text{ mL}}{\text{g Sample}}$$

12.3 ICV, and CCV Recovery:

$$\frac{\text{Actual mg/L}}{0.50\text{ mg/L}} \times 100 = \%\text{ Recovery}$$

12.4 Matrix Spike Recovery:

$$\frac{(\text{Spiked sample conc., mg/L}) - (\text{unspiked sample conc., mg/L})}{0.25\text{ mg/L}} \times 100 = \%\text{ Recovery}$$

12.5 Relative Percent Difference:

$$\frac{|(\text{Sample 1} - \text{Sample 2})|}{\text{Ave., Sample 1 \& 2}} \times 100 = \%\text{RPD}$$

13.0 ACCEPTANCE CRITERIA PHOSPHORUS ANALYSIS:

13.1

ICB, CCB	< 0.02 mg/L
ICV, CCV	85% - 115% Recovery, EXCEPTION: For NC wastewater samples must recover within 90-110%.
Matrix spike	75% - 125% Recovery
Duplicates	$\pm$ 20 % or $\pm$ (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.02 mg/L or current MDL (water); 20 mg/kg (soil) on dry weight basis

Units of Measure: mg/L (water) or mg/kg (soil)

Significant Figures: 2 figures

14.2 Data Reporting:

Below quantitation limit: BQL

QC information included in report:

Level 1 QC:	Results, Blank values, sampling COC.
Level 2 QC:	Results, Blank values, Duplicate data (%RPD), sampling COC.
Level 3 QC:	Results, Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples, sampling and internal laboratory COCs.

15.0 <u>SUPPLEMENTAL DOCUMENTS</u>

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for pH, IEA Doc# CVS02400.NC

15.3 SOP for Soil Homogenization and Percent Solids, IEA Doc# QAS01400.NC.

15.4 SOP for Calibration of Lab Balances, IEA Doc# QAS01002.NET.

16.0 <u>REFERENCES</u>

16.1 Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-P B.

16.2 Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-P E.

16.3 Methods for Chemical Analysis of Water and Waste, 1983, EMSL, EPA-600/4-79-020, Method 365.2.

17.0 <u>SUBSTANTIVE REVISIONS</u>

17.1 Original issue. (07/01/92)

17.2 Addendum to Section 6.4 to clarify holding time. Amendment to Section 8.1.1 from 18.0 to 16.6 meg-ohm reagent water. Section 12.4 amended to more clearly express absolute value. (08/23/96)

17.3 Header corrected to include (W&S). Section 3.3.8, 6.3, 7.7, 8.1.12, 8.2.5, 10.6, 11.2.2, 12.2, 15.1-15.3, 17.2 added. Section 3.2 revised to reflect current method reference. Sections 7.7 - 7.8 added. Section 8.0 revised for clarification. Calibration points revised in Section 8.2.2.1. Section 8.2.3 revised: new voluette concentration for ICV preparation. Section 9.4 revised for correleation coefficient. Sections 9.6 and 9.7 added. Sections 10.2 and 14.1 revised to reflect the current PQLs for water and soils. Sections 10 and 11 amended for clarification. NC wastewater acceptance criteria for ICV/CCV acceptance added in Section 13.1. The units for soil reporting included in Section 14.1. Sections 15.1 - 15.4 added. Attachments added. (04/28/97)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
			R			
			R			
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			R			

Secondary Review By:____________________ Date:__________

IEA, Inc. Form# CVL01200.NC

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____________ Date:_____________

Attachment III

SPECTROPHOTOMETRIC ANALYSIS

Method Name: ____________________ Date: ____________________ Sample Matrix: ____________________

Test Code: ____________________ Prep. Date: ____________________ Analyst: ____________________

Daily Standards Preparation							
Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV							
LCS/QC Check							
Working Standard							
Spike							

STANDARD CURVE

Samples/Standards Final Volume Required: ____________ mL (B)

Volume Std Used	Std #	Std Concentration	Absorbance Reading

Curve Correlation Coefficient (must be $\geq$ 0.995): ____________________ Y-Intercept: ____________________

Sample ID	Volume Used (mLs) (C)	Absorbance	Curve Conc. ug/mL (A)	Final Conc. (ug/mL)	QC Sample (Recovery/RPD)
ICV					
ICB					
LCS/QC CHECK					
CCV					
CCB					

CALCULATIONS:

Concentration = ug/mL = (A) (B/C)

$$RPD = \frac{|\text{Sample} - \text{Sample Dup.}|}{\text{Avg., Sample + Dup.}} \times 100$$

$$\%\ Recovery = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Concentration}} \times 100$$

Comments: ____________________

Spike Witness: ____________________ Date: ____________

Secondary Review By: ____________________ Date: ____________

31. IEA SOP FOR ORTHOPHOSPHATE ANALYSIS FOR WATER AND SOIL

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director John Willey

Quality Assurance Manager [illegible] M. Patterson

2.0 SCOPE AND APPLICATION

2.1 This method describes the procedure for the analysis of aqueous samples for orthophosphate.

2.2 This procedure is applicable to drinking, surface, saline, and ground waters, domestic and industrial wastes and soils and sludges..

2.3 This procedure is applicable to Test Codes OPO4_1 and OPO4_2.

3.0 SUMMARY OF METHOD

3.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test.

3.2 This procedure is based on EPA Method 365.2 and on Standard Methods, 18th Edition, Method 4500-P E.

3.3 This procedure deviates from the above methods as follows:

3.3.1 A 50 ml aliquot of sample or leachate is colored.

3.3.2 Antimony potassium tartrate is stored in a dark bottle at 4 °C. Ammonium

3.3.3 Phosphorus stock solution is 500 mg/L; intermediate standard is 50 mg/L; working standard is 2.5 mg/L.

4.0 INTERFERENCES

4.1 High iron concentrations can cause precipitation of and subsequent loss of phosphorus. Dilution of sample may be necessary to reduce iron interference.

4.2 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination.

4.3 Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

5.0 SAFETY

5.1 Concentrated sulfuric acid can cause severe burns. Goggles or face shield, lab coat, and gloves are necessary when using this reagent. Gloves are recommended when using dilute solutions of sulfuric acid.

5.2 Sodium hydroxide can cause severe burns. Goggles or face shield, lab coat, and gloves are recommended when preparing solutions of this reagent. Gloves are recommended when using dilute solutions of sodium hydroxide.

5.3 Gloves are recommended when handling antimony potassium tartrate, ammonium molybdate, and ascorbic acid; avoid inhalation and contact with skin.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Water samples must be preserved with sulfuric acid (2 ml/liter of sample) to pH less than 2 at time of collection. Samples are stored at 4 degrees C until analysis.

6.3 Soil/sludge samples are cooled to 4 degrees C at time of sampling for transport and storage until analysis.

6.4 The holding time for ortho-phosphate analysis is 48 hours from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Spectronic 1201 spectrophotometer; 1 cm square cuvet

7.2 250 mL beakers, funnels, 100 mL volumetric flasks, 500 mL and 1 Liter volumetric flasks and 125 mL Erlenmeyer flasks pre-rinsed with 50% hydrochloric acid and reagent water.

7.3 #40 filter paper

7.4 Oxford pipetter, 5-10 mL

7.5 Assorted beakers, graduated cylinders, transfer pipets, volumetric pipets, scoopulas

7.6 Top loading balance, 0.1g sensitivity.

7.7 pH Meter

7.8 Stir plate

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm-cm or higher, deionized.

8.1.2 Concentrated sulfuric acid (H_2SO_4), ACS grade.

8.1.2.1 11N H_2SO_4: Carefully add 310 mL concentrated H_2SO_4 to 500 mL reagent water in a 1-liter volumetric flask; swirl to mix; cool and dilute to volume.

8.1.2.2 1N H_2SO_4: Add 28 mL concentrated H_2SO_4 to 900 mL reagent water in a 1000 mL volumetric flask; swirl to mix; cool and dilute to mark.

8.1.2.3 0.02N H_2SO_4: Dilute 5 mL of 1N H_2SO_4 to 250 mL with reagent water.

8.1.3 Sodium hydroxide (NaOH), ACS grade.

8.1.3.1 6N NaOH: Dissolve 120 g NaOH pellets in reagent water; cool and dilute to 500 mL.

8.1.3.2 1N NaOH: Dissolve 40 g NaOH pellets in reagent water; cool and dilute to 1 Liter.

8.1.3.3 0.02N NaOH: Dilute 5 mL of 1N NaOH to 250 mL with reagent water.

8.1.4 Antimony potassium tartrate [$K(SbO)C_4H_4O_6$: ½ H_2O], ACS grade.

8.1.4.1 Antimony potassium tartrate: Dissolve 1.3715 g in reagent water; dilute to 500 mL. Store at 4 °C in a dark bottle.

8.1.5 Ammonium molybdate [$(NH_4)_6Mo_7O_{24}$: 4 H_2O], ACS grade.

8.1.5.1 Ammonium molybdate: Dissolve 20 g in 500 mL reagent water. Store at 4 °C in a plastic bottle.

8.1.6 L-ascorbic acid, ACS grade

8.1.6.1 Ascorbic acid: Dissolve 1.76 g in 100 mL reagent water. Prepare fresh at time of analysis.

8.1.7 Combined color reagent: Mix, in order, per 100 mL of reagent:

50 mL 5N H_2SO_4 (Section 8.1.2.2)
5 mL antimony potassium tartrate (Section 8.1.4.1)
15 mL ammonium molybdate (Section 8.1.5.1)
30 mL ascorbic acid (Section 8.1.6.1)

8.1.8 Mix after addition of each reagent. Reagents should be at room temperature before mixing and must be mixed in the order given. Prepare fresh at time of analysis.

8.1.9 All reagent receipt and preparation is documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standards

8.2.1 Stock/Intermediate/Working Standards

8.2.1.1 Potassium dihydrogen phosphate (KH_2PO_4), dried at 105 °C, ACS grade.

8.2.1.1.1 Stock phosphorus solution, 500 mg/L: Dissolve 1.0985 g dried KH_2PO_4 in reagent water; dilute to 500 mL. Prepare annually.

8.2.1.1.2 Intermediate standard, 50 mg/L: Dilute 10.0 mL of Stock to 100 mL in reagent water. Prepare semi-annually.

8.2.1.1.3 Working standard, 2.5 mg/L: Dilute 50.0 mL of Intermediate standard to 1000 mL in reagent water. Prepare monthly.

8.2.2 Calibration Standards

8.2.2.1 Calibration standards are prepared by the dilution of Working Standard (Section 8.2.1.1.3) to 100 mL in reagent water according to the following table:

Volume of Working Standard (2.5 mg/L)	Calibration Standard Concentration, mg/L
0	0
0.8	0.020
5.0	0.125
15.0	0.375
25.0	0.625
40.0	1.000

8.2.3 Initial and Continuing Calibration Verification Standards (ICV & CCV)

8.2.3.1 ICV/CCV Stock, 25 mg/L, 10 mL voluette, purchased from Hach, Cat.# 21092-10.

8.2.3.2 ICV/CCV Standard, 0.50 mg/L: Empty two 10 mL voluettes (Section 8.2.3.1) into a 1000 mL volumetric flask containing 900 mL reagent water and dilute to volume with reagent water.

8.2.4 Matrix Spike Standard

8.2.4.1 The intermediate standard (Section 8.2.1.1.2) is used to prepare a matrix spike standard at a concentration of 0.25 mg/L as in Section 10.3.

8.2.4.1.1 Pipet 250 uL of the intermediate standard (50 mg/L) into a 50 mL aliquot of water sample prior to addition of color reagent, resulting in a 0.50 mg/L spike concentration.

8.2.4.1.2 Pipet 500 uL of the intermediate standard at 50 mg/L into a 10 g sample prepared for leaching, resulting in a 2.5 mg/Kg spike concentration.

8.2.5 All standard receipt and preparation is documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 Set the absorbance on the Spectronic 1201 at zero using the zero calibration standard (Section 8.2.2.1) and record the absorbance in the Spectrophotometric Analysis Logbook (Attachment III).

9.2 Analyze the calibration standards in succession from the lowest to the highest concentration (Section 8.2.2.1) and record the absorbances in the analysis logbook.

9.3 Correlation coefficients are calculated using linear regression.

9.4 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. concentration to be equal to or greater than 0.995.

9.5 The working calibration range of this method is 0.02 mg/L to 1.00 mg/L for waters and 0.2 mg/Kg to 10 mg/Kg for soils/sludges.

9.6 Calibrate the pH meter to be used for pH adjustment according to the SOP for pH, IEA Doc #CVS02400.NC.

9.7 Calibrate the balance daily prior to use, according to the SOP for Calibration of Lab Balances, IEA Doc #QAS01002.NET.

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The Practical Quantitation Limit (PQL) for this procedure is the MDL, currently 0.02 mg/L for waters and 0.2 mg/kg for soils.

10.3 Matrix spikes are prepared as in Section (8.2.4) and should be performed one per matrix per 10 samples or less being analyzed.

10.3.1 Matrix spike recoveries are determined as in Section 12.4 and should meet the criteria established in Section 13. Recoveries that do not meet this criteria may be attributable to the sample matrix effect.

10.4 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively). ICV/CCVs are prepared by processing 100 mL of ICV/CCV Standard, 0.50 mg/L (Section 8.2.3.2) as a sample.

10.4.1 Recovery calculations and criteria are found in Sections 12 and 13 and must be met.

10.5 Blanks, including the ICB, CCB and Preparation blank, are prepared by processing 100 mL of reagent water as a sample.

10.5.1 Failure to meet acceptance criteria as outlined in Section 13 will require reanalysis for samples associated with the ICB/CCB. Sample will require repreparation and reanalysis for failure of the Preparation blank.

10.6 Internal standards and surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 10 samples, per batch, per matrix
Duplicate	Every 10 samples, per batch

10.7.1 DEFINITIONS:

Batch: Every 10 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 Turn on the Spectronic 1201 and allow the instrument to complete the Self-Check.

11.1.2 Set the wavelength to 880 nm and allow the instrument to warm up for at least 30 minutes.

11.1.3 Place the square cuvet holder in the chamber.

11.2 Sample Preparation

11.2.1 Water samples

11.2.1.1 Pour approximately 150 mL of sample into a pre-rinsed 250 mL beaker. Add stir bar, place beaker on stir plate, and place pH electrodes in solution.

11.2.1.2 Adjust pH of solution to between 6.8 and 7.2, using 6 N, 1 N and 0.02 N NaOH, and 1 N and 0.02 N H_2SO_4, as necessary.

11.2.1.3 Remove beaker from pH meter. If sample has turned golden-yellow and/or formed floc during pH adjustment, add 11 N H_2SO_4 dropwise until color dissipates and/or floc redissolves.

11.2.1.4 Check the sample for turbidity or solid matter and filter, if necessay, through #40 filter paper into another pre-rinsed 250 mL beaker.

11.2.2 Soil/Sludge samples

11.2.2.1 Weigh 10 g of sample into a pre-rinsed 250 mL beaker. Record the weight in the Spectrophotometric Analysis Logbook (Attachment III).

11.2.2.2 Add 100 mL reagent water and stir bar; stir 30 minutes on a stir plate.

11.2.2.3 Adjust the pH of the leachate to between 6.8 and 7.2, using 6N, 1N or 0.02 N NaOH and 1N and 0.02N H_2SO_4, , as necessary.

11.2.2.4 If sample has turned golden yellow and/or formed a floc during pH adjustment, add 11N H_2SO_4 dropwise until color dissipates and/or floc redissolves.

11.2.2.5 Allow leachate to settle. Filter at least 75 mL of leachate through a #40 filter into another pre-rinsed 250 mL beaker.

11.2.3 For ICB, add 150 mL of reagent water to a labeled beaker. Adjust pH as above.

11.2.4 For ICV, add 150 mL of ICV (Section 8.2.3.2) solution to labeled beaker. Adjust pH as above.

11.2.5 For CCV, add 150 mL of ICV solution to labeled beaker. Adjust pH as above.

11.3 Analytical Procedure

11.3.1 Transfer 50 mL of prepared solution into a pre-rinsed 125 mL Erlenmeyer flask. Add 8 mL of combined color reagent and swirl to mix well.

11.3.2 After at least 10 minutes but no more than 30 minutes, set absorbance on the Spectronic 1201 at zero using the 0 mg/L calibration standard.

11.3.3 Read each solution in succession, starting with the lowest concentration calibration standards; record the corresponding absorbance in the analysis logbook.

11.3.3.1 If sample absorbance exceeds the range of the curve, dilute another aliquot of the sample and re-color as above.

12.0 CALCULATIONS:

12.1 Analyte:

Waters

$$\text{mg/L from curve} \times \frac{100\ \text{mL}}{\text{mL sample}} = \text{mg/L as P}$$

Soils

$$\text{mg/L from curve} \times \frac{100\ \text{mL}}{\text{g sample}} = \text{mg/Kg as P}$$

12.2 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{0.50\text{ mg/L}} \times 100 = \%\text{ Recovery}$$

12.3 Spike Recovery:

$$\frac{[\text{Spiked sample conc., mg/L(mg/Kg)}] - [\text{unspiked sample conc., mg/L(mg/Kg)}]}{0.25\text{ mg/L (2.5 mg/Kg)}} \times 100 = \%\text{ Recovery}$$

12.4 Method blank, Initial Calibration and Continuing Calibration blanks: Not to exceed 0.02 mg/L (MDL).

12.5 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1 - Sample 2})}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\text{ RPD}$$

13.0 ACCEPTANCE CRITERIA PHOSPHORUS ANALYSIS:

13.1	ICB, CCB	< 0.02 mg/L
	ICV, CCV	85% - 115% Recovery; **EXCEPTION**: NC regulatory reports require 90-110% Recovery.
	Matrix spike	75% - 125% Recovery
	Duplicates	± 20 % or ± 0.02 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.02 mg/L - waters (or current MDL).
0.2 mg/Kg - soils

Units of Measure: mg/L (waters); mg/Kg (soils)

Significant Figures: 2 figures

Data Reporting:

Below quantitation limit: BQL

14.2 QC information included in report:

Level 1 QC: Results and Blank values, sampling COC.

Level 2 QC: Results, Blank values, Duplicate data (%RPD), sampling COC.

Level 3 QC: Results, Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples, sampling and internal laboratory COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for pH, IEA Doc# CVS02400.NC

15.3 SOP for Soil Homogenization and Percent Solids, IEA Doc# QAS01400.NC.

15.4 SOP for Calibration of Lab Balances, IEA Doc# QAS01002.NET.

16.0 REFERENCES

16.1 Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-P E.

16.2 Methods for Chemical Analysis of Water and Waste, 1983, EMSL, EPA-600/4-79-020, Method 365.2.

17.0 SUBSTANTIVE REVISIONS

17.1 Original issue. (07/01/92)

17.2 Amendment made to Section 2.1. Revision made to Section 3.1. Amendment made to Section 3.3. Addendum to Section 7.2. Amendments made to Sections 8.1 and 8.2. Amendment to 10.1. Revisions to Sections 11.2.1 and 11.2.2. Amendments to Sections 11.2.5 and 11.3.3. Revisions to Section 12.1 and Section 12.2. Amendment to Section 12.5. Amendment to Section 13.0. Amendment to Section 16.2. (09/07/94)

17.3 Header corrected to include (W&S). Sections 2.2, 2.3, 3.3.1, and 4.1 revised. Sections 6.3, 7.6, 7.7, and 7.8 added. Section 8 revised and clarified. Section 9.4 correlation coefficient revised. Section 9.5 revised to include soil range. Section 9.6 added. Sections 10 and 11 revised and clarified. Section 12.3 revised for soil calculation. Sections 13 and 14 revised for soil units. Section 13 revised to include NC regulatory criteria. Sections 15.1 - 15.4 added. Section 16 revised to proper format. Attachments added. (04/29/97)

ATTACHMENT I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:__________________ Date:__________

IEA, Inc. Form# CVL01200.NC

ATTACHMENT II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:____________ Date:____________

ATTACHMENT III

SPECTROPHOTOMETRIC ANALYSIS

Method Name: ____________ Date: ____________ Sample Matrix: ____________
Test Code: ____________ Prep. Date: ____________ Analyst: ____________

Daily Standards Preparation							
Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV							
LCS/QC Check							
Working Standard							
Spike							

STANDARD CURVE

Samples/Standards Final Volume Required: ____________ mL (B)

Volume Std Used	Std #	Std Concentration	Absorbance Reading

Curve Correlation Coefficient (must be ≥ 0.995): ____________ Y-Intercept: ____________

Sample ID	Volume Used (mLs) (C)	Absorbance	Curve Conc. ug/mL (A)	Final Conc. (ug/mL)	QC Sample (Recovery/RPD)
ICV					
ICB					
LCS/QC CHECK					
CCV					
CCB					

CALCULATIONS:

Concentration = ug/mL = (A) (B/C)

RPD = |(Sample - Sample Dup.)| / Avg., Sample + Dup. X 100

% Recovery = Spiked Sample Result - Sample Result / Spike Concentration X 100

Comments: ____________

Spike Witness: ____________ Date: ____________

Secondary Review By: ____________ Date: ____________

32. IEA SOP FOR TOTAL KJELDAHL NITROGEN

33. IEA SOP FOR NITRATE/NITRITE ANALYSIS

34. IEA SOP FOR AMMONIA ANALYSIS

35. IEA SOP FOR CHLORIDE IN WATER

36. IEA SOP FOR FLUORIDE ANALYSIS

37. IEA SOP FOR SULFATE IN WATER

38. IEA SOP FOR SULFIDE ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director ____________________

Quality Assurance Manager ____________________

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This procedure is applicable to the measurement of total and dissolved sulfides in drinking surface and saline waters, domestic and industrial wastes as well as soil sample leachates. Excess iodine is added to a sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate or phenylarsine oxide. This procedure is suitable for the measurement of sulfide in concentrations above 1 mg/L.

2.2 Acid insoluble sulfides are not measured by this test. (Copper sulfide is the only common sulfide in this class.

2.3 This procedure is applicable to Test Codes S_01 & S_02.

3.0 SUMMARY OF METHOD

3.1 200 mL or an aliquot of sample diluted to 200 mL is placed in a 250 mL erlenmeyer flask. 2 mL of 6N HCl and 10 mL of 0.025N iodine solution are then added to the flask. The solution in the flask is titrated with sodium thiosulfate solution until a pale yellow color is obtained. 6-8 drops of starch indicator is then added to the erlenmeyer flask resulting in a dark blue solution. The solution in the erlenmeyer is backtitrate with sodium thiosulfate solution until the blue color is no longer visible. Initial and final volumes of titrant are recorded to determine the mg/L of sulfide present.

3.2 This procedure is based on EPA Method 376.1.

3.3 This SOP deviates from EPA Method 376.1 in the following manner:

3.3.1 This SOP is modified to accommodate the analysis of soils.

4.0 INTERFERENCES

4.1 Reduced sulfur compounds, such as sulfite, thiosulfate and hydrosulfite which decompose in acid may yield erratic results. Also, volatile iodine-consuming substances will give high results.

4.2 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

4.3 If the sample is not preserved with zinc acetate, the analysis must be started immediately. Similarly, the measurement of dissolved sulfides must be also begin immediately.

5.0 SAFETY

5.1 The use of sodium thiosulfate solution, and 6N HCL acid solution may cause skin irritation. Safety glasses, lab coat, and latex gloves are necessary to prevent contact with these solutions.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers must be glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample collection: Samples are collected with zinc acetate as a preservative and stored at 4 °C until analysis. Samples are shipped to the laboratory at 4 °C and stored at 4 °C until analysis.

6.3 Sample Preservation

6.3.1 Water samples are preserved with 2 mL zinc acetate and NaOH to a pH > 9.

6.3.2 Soil samples are cooled to 4 °C until analysis.

6.4 The holding time for sulfide analysis is 7 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 25 mL buret.

7.2 250 mL Erlenmeyer flasks.

7.3 10 mL pipet.

7.4 250 mL graduated cylinder.

7.5 Magnetic stirrer, stir bar.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm or higher, deionized.

8.1.2 Concentrated Hydrochloric Acid, purchased from a commercial vendor.

8.1.2.1 6N HCl: Carefully dilute 125 mL of concentrated HCl (Section 8.1.2) to 250 mL with reagent water (Section 8.1.1).

8.1.3 Iodine solution, 1N, purchased from a commercial vendor.

8.1.3.1 0.025N iodine solution: Dilute 25 mL 1N iodine solution (Section 8.1.3) to 1000 mL with reagent water (Section 8.1.1).

8.1.4 Soluble starch, purchased from a commercial vendor.

8.1.5 Salicylic acid, purchased from a commercial vendor.

8.1.6 Starch indicator solution: Dissolve 5 g starch (Section 8.1.4) and 0.5 g salicylic acid into 200 mL of hot reagent water with constant stirring until dissolved. Dilute to a final volume of 250 mL with reagent water (Section 8.1.1).

8.1.7 All reagent receipt and preparation is documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standard solutions

8.2.1 Sodium Thiosulfate solution, 0.025N NaS_2O_3 certified lot analysis from Fisher Scientific Co., (Cat. no. SS370.1).

8.2.2 All standard receipt and preparation is documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 Calibration is not applicable.

9.2 The working range of this method is 1 mg/L and above. Samples requiring in excess of 50 mL of standard titrant are diluted within range.

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The practical quantitation limit (PQL) is 1 mg/L for water samples and 1 mg/kg for soil samples.

10.3 Matrix spikes are not practical for this method.

10.4 QC check samples consist of Initial and Continuing Blanks. Blanks are performed at the onset of analysis and after every ten samples.

10.4.1 Blanks are prepared by aliquoting 200 mL of reagent water and analyzing as other samples.

10.5 Surrogates are not applicable to this method.

10.6 Duplicate samples are analyzed every 10 samples.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Duplicate Samples	Every 10 samples, per analysis

10.7.1 Definitions

Batch: Every 10 samples, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Water Samples

11.1.1 Rinse a clean 25 mL buret three times with titrant (sodium thiosulfate, Section 8.2.1). Fill the buret, making sure that no air bubbles are present.

11.1.2 Add 200 mL of sample to a 250 mL erlenmeyer flask. If the sample appears to be turbid or cloudy, a smaller aliquot may be diluted to 200 mL. Record the initial volume of sample used in the Titrimetric Analysis Logbook (Attachment III).

11.1.3 Add 10.0 mL of 0.025N iodine solution (Section 8.1.3.1) and 2 mL of 6N HCl (Section 8.1.2.1).

11.1.4 Record the initial volume of titrant in the Titrimetric Analysis Logbook.

11.1.5 Titrate the solution in the erlenmeyer with sodium thiosulfate until a pale yellow color is achieved. Record the final volume of titrant in the Titrimetric Analysis Logbook.

11.1.6 Add 6-8 drops of starch indicator (Section 8.1.6). The solution in the erlenmeyer should be dark blue.

11.1.7 Record the initial volume of titrant in the Titrimetric Analysis Logbook.

11.1.8 Titrate the solution in the erlenmeyer with sodium thiosulfate until the solution is clear. Record the final volume of titrant in the Titrimetric Analysis Logbook.

11.2 Soil Samples

11.2.1 Weigh 25 g of soil sample into an erlenmeyer flask and add 250 mL of reagent water. Let the erlenmeyer stand for 20 minutes. Record the weight of sample leached in the Titrimetric Analysis Logbook.

11.2.2 Decant the supernatant and centrifuge. Pour off 200 mL of the centrifuged solution into a 250 mL erlenmeyer flask and titrate as in Sections 11.1.3 - 11.1.8.

11.3 Samples Containing Precipitate

11.3.1 Samples with precipitate should be titrated directly in the original bottle as in Sections 11.1.3 - 11.1.8.

11.3.2 When the titration is complete, transfer the contents of the sample bottle to a graduated cylinder to determine the original sample volume (subtract the volume of titrant added).

12.0 CALCULATIONS

12.1 Water Samples

$$\text{mg sulfide/L} = \frac{[(ab)-(cd)] \times 16000}{\text{volume of sample (mL)}}$$

a = mL of iodine solution (10 mL)
b = Normality of iodine solution (0.025N)
c = mL of sodium thiosulfate used
d = Normality of sodium thiosulfate (0.025N)

12.2 Soil Samples

$$\text{mg sulfide/kg} = \frac{[(ab)-(cd)] \times 16000 \times 250\text{ mL}}{\text{wt of sample leached (g)}}$$

a = mL of iodine solution (10 mL)
b = Normality of iodine solution (0.025N)
c = mL of sodium thiosulfate used
d = Normality of sodium thiosulfate (0.025N)

12.3 Relative Percent Difference

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1	ICB, CCB	< PQL
	DUPLICATES	+/- 20% or +/-1 mg if sample conc. <5 x MDL.

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limit: 1.0 mg/L for waters and 1.0 mg/kg for soils
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level I QC: Blank values, Sampling COCs.
Level II QC: Blank values, Duplicate data (%RPD), Sampling COCs.
Level III QC: Blank values, Duplicate data (%RPD) and Sampling and Laboratory COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Methods for Chemical Analysis of Water and Waste, 1983, EMSL, EPA-600/4-79-020. Method 376.1.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue

17.2 Section 8.1.1 revised from 18.0 megohm distilled water to 16.6. Section 11.2.5 added to address precipitated samples. (04/18/95)

17.3 Section 2.0 revised for clarification. Sections 3.3, 6.3.2, 10.4.1, and 12.2 added. Section 8.0 revised for clarification. Duplicate frequency added in Section 10.7. Section 12.1 revised. Sections 10.2 and 14.1 revised to reflect current PQLs for soil and water samples. Section 12.2 added. (08/08/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:____________________ Date:__________

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:______________ Date:______________

Attachment III

TITRIMETRIC ANALYSIS

Method Name:________________ DATE:________________ Analyst:________________

Daily Standards and Spike Preparation

Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV/QC Check							
Working Standard							
Spike							

TITRANT:________________ Normality:____________ Reference:________________

Sample ID	mLs Sample	Titrant V_i	Titrant V_e	Titrant V_T	ug/mL	% Rec. / RPD
ICV						
ICB						

CALCULATIONS:

RPD = | (Sample - Sample Dup.) | X 100 / Avg., Sample + Dup.

% Recovery = Spiked Sample Result - Sample Result X 100 / Spike Concentration

Concentration:

ug/mL =

meq wt =

Comments:________________________________

Spike Witness By:________________ Date:____________

Secondary Review By:________________ Date:____________

39. IEA SOP FOR ACIDITY ANALYSIS

40. IEA SOP FOR pH IN WATER

41. IEA SOP FOR pH IN SOIL

42. GAILBRAITH LABORATORIES SOP FOR TOTAL SULFUR

Designation: D 4239 – 85

Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods [1]

This standard is issued under the fixed designation D 4239; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover three alternative procedures using high-temperature tube furnace combustion methods for the rapid determination of sulfur in samples of coal and coke.

1.2 These test methods appear in the following order:

	Sections
Method A—High Temperature Combustion Method with Acid Base Titration Detection Procedures	6 to 9
Method B—High Temperature Combustion Method with Iodimetric Titration Detection Procedures	10 to 13
Method C—High Temperature Combustion Method with Infrared Absorption Detection Procedures	14 to 16

1.2.1 When automated equipment is used to perform any of the three methods of this test method, the procedures can be classified as instrumental methods. There are several manufacturers that offer to the coal industry equipment with instrumental analysis capabilities for the determination of the sulfur content of coal and coke samples.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See 7.8 and 15.2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 1193 Specification for Reagent Water[3]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 2361 Test Method for Chlorine in Coal[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 3176 Practice for Ultimate Analysis of Coal and Coke[2]

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

D 4208 Test Method for Total Chlorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method[2]

3. Summary of Methods

3.1 *Method A—High-Temperature Combustion Method with Acid-Base Titration Detection Procedures*—A weighed sample is burned in a tube furnace at a minimum operating temperature of 1350°C in a stream of oxygen. During combustion, all sulfur contained in the sample is oxidized to gaseous oxides of sulfur (sulfur dioxide, SO_2, and sulfur trioxide, SO_3) and the chlorine in the sample is released as Cl_2. These products are then absorbed into a solution of hydrogen peroxide (H_2O_2), where they dissolve forming dilute solutions of sulfuric (H_2SO_4) and hydrochloric (HCl) acids. The quantities of both acids produced are directly dependent upon the amounts of sulfur and chlorine present in the original coal sample. Once the amounts of each acid present have been determined, the percentage of sulfur contained in the coal may be calculated.

3.1.1 This method is written to include commercially available sulfur analyzers, that must be calibrated with appropriate standard reference materials (SRM's), in order to establish recovery factors or a calibration curve based on the range of sulfur in the coal or coke samples being analyzed.

NOTE 1—Elements ordinarily present in coal do not interfere in Method A (3.1), with the exception of chlorine; results must be corrected for chlorine content of the samples (9.1).

3.2 *Method B—High Temperature Combustion Method with Iodimetric Detection Procedures*—A weighed sample is burned in a tube furnace at a minimum operating temperature of 1350°C in a stream of oxygen to ensure the oxidation of sulfur. The combustion products are absorbed in an aqueous solution that contains iodine. When sulfur dioxide is scrubbed by the diluent, the trace iodine originally present in the solution is reduced to iodide, thus causing an increase in resistance. The detection system of the instrument consists of a polarized dual platinum electrode. Any change in resistance of the solution in the vessel is detected. Iodine titrant is then added proportionally to the reaction vessel until the trace excess of iodine is replenished and the solution resistance is reduced to its initial level. The volume of titrant expended is used to calculate the sulfur concentration of the sample. The method is empirical; therefore, the apparatus must be calibrated by the use of standard reference material (SRM).

3.2.1 This method is designed to be used with commercially available sulfur analyzers, equipped to perform the preceding operation automatically, and must be calibrated

[1] This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved Nov. 29, 1985. Published January 1986. Originally published as D 4239 - 83. Last previous edition D 4239 - 83.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

with an appropriate sample (5.4) based on the range of sulfur in each coal or coke sample analyzed.

NOTE 2—Nonautomatic systems may be used with the titration procedures and calculations performed manually by qualified laboratory technicians. The resulting loss in accuracy or speed, or both, would then negate the advantages of using the fully automated instrumental approach.

3.3 *Method C—High Temperature Combustion Method with Infrared Absorption Detection Procedures*—The sample is burned in a tube furnace at a minimum operating temperature of 1350°C in a stream of oxygen to oxidize the sulfur. Moisture and particulates are removed from the gas by traps filled with anhydrous magnesium perchlorate. The gas stream is passed through a cell in which sulfur dioxide is measured by an infrared (IR) absorption detector. Sulfur dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy is absorbed as the gas passes through the cell body in which the IR energy is being transmitted: thus, at the detector, less energy is received. All other IR energy is eliminated from reaching the detector by a precise wavelength filter. Thus, the absorption of IR energy can be attributed only to sulfur dioxide whose concentration is proportional to the change in energy at the detector. One cell is used as both a reference and a measurement chamber. Total sulfur as sulfur dioxide is detected on a continuous basis. This method is empirical; therefore, the apparatus must be calibrated by the use of standard reference materials (SRM).

3.3.1 This method is for use with commercially available sulfur analyzers equipped to carry out the preceding operations automatically and must be calibrated using standard reference material (coal) of known sulfur content based on the range of sulfur in each coal or coke sample analyzed.

4. Significance and Use

4.1 Determination of sulfur is, by definition, part of the ultimate analysis of coal.

4.2 Results of the sulfur analysis are used to serve a number of interests: evaluation of coal preparation, evaluation of potential sulfur emissions from coal combustion or conversion processes, and evaluation of the coal quality in relation to contract specifications, as well as other scientific purposes.

4.3 The instrumental analysis provides a reliable, rapid method for determining the concentration of sulfur in a lot of coal or coke and are especially applicable when results must be obtained rapidly for the successful completion of industrial, beneficiation, trade, or other evaluations.

5. Sample

5.1 The sample shall be the material pulverized to pass No. 60 (250-μm) sieve and mixed thoroughly in accordance with Method D 2013 or Method D 346.

NOTE 3—It may be difficult to meet the precision statements of Section 19 when high mineral content coals are ground to pass 60 mesh. When the precision of analysis required cannot be obtained, it is recommended that the coals be ground to pass through a No. 100 (150-μm) sieve. The reduced particle size should result in a more homogeneous sample.

5.2 A separate portion of the analysis sample should be analyzed for moisture content in accordance with Test Method D 3173, so that calculation to other than the as-determined basis can be made.

5.3 Procedures for converting as-determined sulfur values obtained from the analysis sample to other bases are described in Method D 3176 and Method D 3180.

5.4 Standard Reference Material (SRM) such as SRM Nos. 2682 through 2685–*Sulfur in Coal*[4] which consist of four different coals that have been individually crushed and ground to pass a 60-mesh sieve, and bottled in 50-g units, or other commercially available reference coals with a certified sulfur content.

METHOD A—HIGH-TEMPERATURE COMBUSTION METHOD WITH ACID-BASE TITRATION DETECTION PROCEDURES[5]

6. Apparatus

6.1 *Tube Furnace*—Capable of heating 150 to 175-mm area (hot zone) of the combustion tube (6.2) to at least 1350°C. It is usually heated electrically using resistance rods, a resistance wire, or molybdenum disilicide elements. Specific dimensions may vary with manufacturer's design.

NOTE 4—Induction furnace techniques may be used provided it can be shown that they meet the precision requirements of Section 19.

6.2 *Combustion Tube*—Approximately 28-mm internal diameter with a 3-mm wall thickness and 750 mm in length made of porcelain, zircon, or mullite. It must be gas-tight at working temperature. The combustion may be carried out in a tapered-end tube that is closely connected to the gas absorber by high temperature tubing with gas-tight joints. Acceptable configurations include connecting the tapered-end tube directly to the elbow of the fritted gas bubbler or to a 10/30 standard taper-ground joint that is attached to a heat resistant glass right angle bend. The temperature at the tapered end of the tube should be maintained high enough to prevent condensation in the tube itself.

6.2.1 Alternatively, a high-temperature straight refractory tube may be used, if available. It requires a silica adaptor (6.11) with a flared end that fits inside the combustion tube and serves as an exit for the gases.

6.3 *Flowmeter*, for measuring an oxygen flow rate up to 2.0 L/min.

6.4 *Sample Combustion Boats*, must be made of iron-free material and of a convenient size suitable for the dimensions of the instrument being used.

6.5 *Boat Puller*—Rod of a heat-resistant material with a bent or disc end to insert and remove boats from the combustion tube.

6.5.1 If the boat puller is to remain within the combustion tube while the boat is moved into the hot zone, it is necessary to pass the puller through a T-piece that is fitted into a rubber stopper at the inlet of the combustion tube. The open end of the T-piece is sealed with a rubber stopper to permit

[4] Available from the Office of Standard Reference Materials, Room B314, Chemistry Bldg, National Bureau of Standards, Washington, DC 20234.

[5] Based on the method of Mott, R. A., and Wilkinson, H. C., "Determination of Sulfur in Coal and Coke by the Sheffield High Temperature Method," *Fuel*, Fuel B, Vol. 35, 1956, p. 6. This method is designed for the rapid determination of sulfur in coal and coke. It is not applicable to coals or coal density fractions that have been subjected to treatment with chlorinated hydrocarbons because of the potentially high acidity of the combustion gases.

where:
T = titrant factor, mg of sulfur/mL,
S_s = sulfur concentration of standard, dry basis,
W' = weight of standard, mg, and
V_t = volume of titrant, mL.

13.2 On analyzers that do not calculate the percent sulfur in the analysis sample automatically, the following calculation must be used:

$$S = 100\,(T \times V_t)/W$$

where:
S = percent sulfur (as determined),
T = titrant factor (see 13.1),
V_t = volume of titrant, mL, and
W' = weight of sample, mg.

METHOD C—HIGH TEMPERATURE COMBUSTION METHOD WITH INFRARED ABSORPTION PROCEDURE

14. Apparatus

14.1 *Measurement Apparatus*—Equipped to automatically combust the sample as described in 3.3. (See Note 8.)

14.2 *Tube Furnace*—See 6.1.

14.3 *Combustion Tube*—Made of mullite, porcelain, or zircon, approximately 40- to 45-mm inside diameter with a 3-mm thick wall, at least 450-mm long with provisions for routing the gasses produced by combustion through the infrared cell.

14.4 *Sample Combustion Boats*—See 6.4.

14.5 *Boat Puller*—See 6.5.

15. Reagents

15.1 *Purity of Reagents*—See 7.1.

15.2 *Magnesium Perchlorate*.—**Warning**: Magnesium perchlorate is a strong oxidizing agent. Do not try to regenerate the absorbent. Do not allow contact with organic materials or reducing agents.

15.3 *Oxygen*—See 7.8.

16. Procedure

16.1 *Instrument Preparation*—Assemble the apparatus according to the manufacturer's instructions. Make a minimum of two determinations (see 16.3) to condition the equipment prior to calibrating the system.

16.2 *Calibration*—Select five coal standard reference materials (SRM) containing known sulfur values of approximately 0.5, 1.0, 2.0, 3.0, and 4.0 % sulfur.

NOTE 10—All SRM used should be prepared in accordance with 5.4.

16.2.1 *Adjustment of Response of Measurement System*—Weigh out approximately 0.5 g of the 3.0 % sulfur standard. Analyze the sample (see 16.3). Repeat this procedure. Adjust instrument as recommended by the manufacturer until the absence of drift is indicated.

16.2.2 *Calibration Procedure*—Weigh out four samples of the 3.0 % sulfur standard (Note 11). Follow the calibration procedure recommended by the manufacturer. Confirm the calibration by analyzing the 3.0 % sulfur standard. The value should be within the allowable limits of the known value. If not, repeat the procedure. Then weigh out and analyze two samples, each of the other calibration standards (Note 11). Record the results after each analysis. Compare the results obtained to the known sulfur values of the samples; they should be within the allowable limits of the known value of the respective sample. If not, refer to the manufacturer's instructions for checking linearity of the analyzer.

NOTE 11—Coal with known sulfur values of less than 2.0 %, use 0.5 g of sample. Coal with known sulfur values greater than 2.0 %, use 0.25 to not more than 0.5 g of sample.

16.3 *Analysis Procedure:*

16.3.1 Stabilize and calibrate the analyzer (see 16.2).

16.3.2 Raise the furnace temperature as recommended by the manufacturer to at least 1350°C. Weigh the sample not more than 0.5 g of coal or 0.25 g of coke (Note 11). Spread the sample evenly in a combustion boat and use a boat puller to position the sample in the hot zone of the furnace for at least 2 min, or until completely combusted.

NOTE 12—The analytical cycle should begin automatically as soon as sulfur is detected.

16.3.3 When the analysis is complete, the instrument should indicate the sulfur value. Refer to the manufacturer's recommended procedure.

17. Report

17.1 The percent sulfur value obtained using any of the described methods is on an as-determined basis.

17.2 The results of the sulfur analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

17.3 Use the percentage of moisture in the sample passing a No. 60 (250-μm) sieve to calculate the as-determined results of the analysis sample to a dry basis.

17.4 Procedures for converting the value obtained on the analysis sample to other bases are described in Method D 3176 and Method D 3180.

18. Precision and Bias

18.1 These are empirical methods that are highly dependent upon the calibration of the equipment, the closeness of the standards to the samples in sulfur content, chlorine content, iron content, etc.

18.2 *Precision Statement for High Temperature Combustion Method Using Acid Base Titration Detection Procedures*—The relative precision of this method for the determination of total sulfur covers the concentration range from 0.5 to 6.0 %.

18.2.1 *Repeatability*—The difference in absolute value between two consecutive test results carried out on the same sample of 60-mesh pulp, in the same laboratory, by the same operator, using the same apparatus, should not exceed the repeatability interval I(r) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one or both of the test results. The repeatability interval may be calculated by use of the following equation:

$$I(r) = 0.06 + 0.03\,\bar{x}$$

where $\bar{x}$ is the average of the two test results.

NOTE 13—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur gave values of 1.52 and 1.57 %. The average sulfur of the duplicate analysis value is 1.55 % and the calculated repeatability I(r) is 0.11. The difference between the two sulfur values is 0.05 and does not exceed the I(r) of 0.11; therefore, these two values are acceptable at the 95 % confidence level.

18.2.2 *Reproducibility*—The difference in absolute value between the averages of replicate determinations, carried out in different laboratories on representative 60-mesh samples, prepared from the same bulk sample after the last stage of reduction, should *not* exceed the reproducibility interval I(R) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one, or both, of the test results. The reproducibility interval may be calculated by the use of the following equation:

$$I(R) = 0.03 + 0.11\,\bar{x}$$

where $\bar{x}$ is the average of between-laboratory results.

NOTE 14—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur in one laboratory gave an average value of 3.81 % and a value of 4.00 % was obtained in a different laboratory. The between laboratory average sulfur value is 3.91 %, the calculated I(R) interval is 0.46 %, and the difference between the different laboratory values is 0.19 %. Since this difference is less than the I(R), these two values are acceptable at the 95 % confidence level.

18.3 *Precision Statement for High Temperature Combustion Method Using Iodimetric Detection Procedures*—The relative precision of this method for the determination of total sulfur covers the concentration range from 0.5 to 6.0 %.

18.3.1 *Repeatability*—The difference in absolute value between two consecutive test results carried out on the same sample of 60-mesh pulp, in the same laboratory, by the same operator, using the same apparatus should not exceed the repeatability interval I(r) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one, or both, of the test results. The repeatability interval may be determined by use of the following equation:

$$I(r) = 0.08\,\bar{x}$$

where $\bar{x}$ is the average of the two test results.

NOTE 15—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur gave values of 1.52 and 1.57 %. The average sulfur of the duplicate analysis value is 1.55 % and the calculated repeatability interval I(r) is 0.12. The difference between the two sulfur values is 0.05 and does not exceed the I(r) of 0.12; therefore, these two values are acceptable at the 95 % confidence level.

18.3.2 *Reproducibility*—The difference in absolute value between the averages of replicate determinations, carried out in different laboratories on representative 60-mesh samples prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility interval I(R) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one, or both, of the test results. The reproducibility interval may be determined by use of the following equation:

$$I(R) = 0.08 + 0.09\,x$$

where x is the average of the between-laboratory results.

NOTE 16—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur in one laboratory gave an average value of 3.81 % and a value of 4.00 % was obtained in a different laboratory. The between-laboratory average sulfur value is 3.91 %, the calculated I(R) interval is 0.43 %, and the difference between the different laboratory values is 0.19 %. Since this difference is less than the I(R), these two values are acceptable at the 95 % confidence level.

18.4 *Precision Statement for High Temperature Combustion Method Using Infrared Absorption Detection Procedures*—The relative precision of this method for the determination of total combustible sulfur covers the concentration range from 0.5 to 6.0 %.

18.4.1 *Repeatability*—The difference in absolute value between two consecutive test results, carried out on the same sample of 60-mesh pulp, in the same laboratory, by the same operator, using the same apparatus should not exceed the repeatability interval I(r) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one, or both, of the test results. The repeatability interval may be determined by use of the following equation:

$$I(r) = 0.03 + 0.04\,\bar{x}$$

where $\bar{x}$ is the average of the two test results.

NOTE 17—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur gave values of 1.52 and 1.57 %. The average sulfur of the duplicate analysis value is 1.55 % and the calculated repeatability interval I(r) is 0.09. The difference between the two sulfur values is 0.05 and does not exceed the I(r) of 0.09; therefore, these two values are acceptable at the 95 % confidence level.

18.4.2 *Reproducibility*—The difference in absolute value between the averages of replicate determinations, carried out in different laboratories on representative 60-mesh samples, prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility interval I(R) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one, or both, of the test results. The reproducibility interval may be determined by use of the following equation:

$$I(R) = 0.05 + 0.06\,\bar{x}$$

where $\bar{x}$ is the average of the between laboratory results.

NOTE 18—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived form the statistical evaluation of the round-robin analytical results. *Example:* Duplicate analysis for total sulfur in one laboratory gave an average value of 3.81 % and a value of 4.00 % was obtained in a different laboratory. The between laboratory average sulfur value is 3.91 %, the calculated I(R) interval is 0.28 %, and the difference between the different laboratory values is 0.19 %. Since this difference is less than the I(R), these two values are acceptable at the 95 % confidence level.

18.5 *Bias*—Bias is eliminated when the instrument is properly calibrated against certified reference standards. Proper calibration includes comparison of instrumental results to certified sulfur values. Results for certified standards above and below anticipated analysis sample results should be within certified precision levels for all standards over the calibration range for the instrument.

REVISION OF APPENDICES B THROUGH D OF THE PILOT/ TREATABILITY TESTING QAPP AND FIELD SAMPLING PLAN

Some of the tables in Appendices B through D were revised in response to the U.S. EPA comments. Tables B-4 through D-9 are provided herein in entirety for easy replacement.

TABLE B-4

CORRECTIVE ACTION SUMMARY FOR SW 846-8270

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8270 GC/MS	Base/Neutral Acid Extractable	Tune the instrument using a deca-fluorotriphenylphosine standard	Every 12 hours	Must meet the ion abundance criteria in SW 846	• Retune instrument, • Repeat standard analysis
		Initial calibration (5 point minimum)	Prior to analysis and as required	% Relative standard deviation for CCC ≤ 30%; Average RF ≥0.05 for SPCC	• Evaluate the system • Repeat calibration
		Continuing calibration check	Every 12 hours	Percent difference for CCC ≤ 30%; RF ≥ 0.05 for SPCC.	• Evaluate system • Reanalyze calibration check standard • Evaluate standard • Repeat the initial calibration as necessary • Reanalyze affected samples
		Method blank	1 per preparation batch (≤ 20 samples)	<PQL; except 5x CRQL for common laboratory contaminants. Surrogate recoveries must be within control limits	• Assess impact on data • Reanalyze blank • Run a system blank if necessary • Reprep/analyze batch as necessary
		Surrogate spike	Every sample, method blank, LCS and MS/MSD	No more than one surrogate per fraction outside of project acceptance criteria; No surrogate below 10% recovery	• Evaluate the system and data • Reanalyze the sample once • Reextract if >1 surrogate per fraction outside acceptance limits • Narrate all outliers
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	• Assess data (4x rule) • Reanalyze once, report both sets • Narrate all outliers
		Matrix spike duplicate	1 per 20 samples	% Recovery and RPD within project QC acceptance criteria	• Same as MS
		Laboratory control sample	1 per preparation batch (≤ 20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	• Reanalyze LCS • Assess impact on data • Reprep/reanalyze batch as needed • Narrate all outliers

TABLE B-5

CORRECTIVE ACTION SUMMARY FOR METHOD SW 846-8081

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8081 GC	Organochlorine Pesticides and PCBs	Initial multipoint calibration	Initially and as required	%RSD ≤ 20% or correlation coefficient (r) ≥ 0.995 or % RSE≤ 20% to accept curve	Recalibrate
		Initial calibration verification (ICV)	Prior to sample analysis	± 15% of expected concentration	Reanalyze ICV Recalibrate Reanalyze affected samples
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 15% of expected concentration for each standard bracketing samples	Reanalyze CCV Reanalyze all affected samples Repeat initial calibration as necessary
		Degradation standard	Every 24 hours of analysis	Breakdown of endrin or 4,4'-DDT < 20%	Evaluate system Perform system maintenance Recalibrate as necessary
		Method blank	1 per preparation batch (≤20 samples)	< RL	Assess impact on data Reanalyze method blank Reprep/reanalyze batch as necessary
		System blank	As required	< RL	Run until system is clean Bake out system Perform maintenance
		Surrogate spike	Every sample, standard, method blank, LCS and MS/MSD	% recovery within acceptance windows for a minimum of one of two surrogate spikes	Assess data Reanalyze sample once Evaluate system Narrate all outliers

TABLE B-5

CORRECTIVE ACTION SUMMARY FOR METHOD SW 846-8081

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8081 GC (continued)		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance limits	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix spike duplicate	1 per 20 samples	% Recovery and RPD within project QC acceptance limits	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	Reanalyze LCS Assess impact on data Reprep/reanalyze batch as necessary Narrate all outliers

TABLE B-6

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8150

Analytical Method	Parameters	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW846, 8150 GC/ECD	Pentachlorophenol	Initial multipoint calibration (5 point minimum)	Initially and as required	%RSD < 20% or correlation coefficient (r) ≥ 0.995	• Evaluate system • Recalibrate
		Initial calibration verification (ICV)	After calibration and prior to sample analysis	± 15% difference from expected concentration	• Evaluate system • Reanalyze ICV • Recalibrate
		Continuing calibration verification (CCV)	Each day and end of run sequence, plus each 10th sample	± 15% difference from expected concentration for each standard bracketing samples	• Evaluate system • Reanalyze CCV • Recalibrate if necessary • Reanalyze affected samples
		Method blank	1 per preparation batch (≤20 samples)	< Reporting Limit	• Assess impact on data • Reanalyze method blank • Reprep/reanalyze batch as necessary
		System blank	As required	< Reporting Limit	• Run until system is clean • Bake out system • Perform maintenance
		Surrogate spike	Every sample, standard, method blank, LCS, and MS/MSD	% Recovery within project QC acceptance criteria	• Assess data • Reanalyze sample once • Evaluate system • Narrate all outliers
		Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	• Assess data (4x rule) • Reanalyze once, report both sets • Narrate all outliers

TABLE B-6

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8150

Analytical Method	Parameters	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW846, 8150 GC/ECD (continued		Matrix spike duplicate (MSD)	1 per preparation batch (≤20 samples)	% Recovery and RPD within project QC acceptance criteria	• Same as matrix spike
		Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	• Reanalyze LCS • Assess impact on data • Reprep/reanalyze batch as necessary • Narrate all outliers

TABLE B-7

DIOXIN/FURAN CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-8290 (GC/MS)	Dioxins/Furans	Tune using PFK	Once per 12 hours, prior to sample analysis	Resolving power ≥ 10,000 at m/z=304.9824 and m/x 380.9760 ≤5 ppm of expected mass	• Retune instrument • Reanalyze PFK
		Window defining mix (WDM) Column Performance Check Solution (CPSM)	Prior to ICAL, once per 12 hours prior to sample analysis	Used to set retention times. CPSM must have ≤25% valley resolution for 2378-TCDD	• Readjust windows • Evaluate system • Perform maintenance • Reanalyze WDN/CPSN
		Multipoint calibration (5 points, ICAL)	Initially and as required	Int std = %RSD≤30% Natives - %RSD≤20% Retention time must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios within Table A limits, and S/N ≥ 2.5	• Evaluate system • Recalibrate
		Daily continuing calibration standard (CCAL)	Once per 12 hours, prior to sample analysis	%D of IS ≤30% from avg RRF (ICAL). %D of natives ≤29% from avg RRD (ICAL). RT must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios are within Table A limits	• Evaluate system • Reanalyze CCAL • Recalibrate (ICAL) as necessary
SW-8290 (GC/HRMS)	Dioxins/Furans	Internal standards	Every sample, method blank, and LCS	Internal standard recovery within limits stated in Tables 2 and 3	• Check chromatogram for interference. If found, flag data • Check instrument and reanalyze the extract if a problem is found and corrected • Check S/W. If <10:1, reextract sample • Evaluate data usability and flag as appropriate • Reextract and reanalyze adversely affected samples
		Method blank	One per analytical batch, not to exceed 20 samples, per matrix	Ratio of a given unlabeled PCDD/PCDF isomer ≤5% of the appropriate internal standard	• Reanalyze method blank • If still exceeds and analyte concentration in sample <CRQL or >10x blank concentration, report results
		LCS (include natives)	At a frequency of 5% (1 per 20 samples analyzed)	Refer to Table 3	• Review Internal Standards, as above • Evaluate data for usability • If sample results are ND and CRQLs are met, no action required
		Duplicates	As per client request	Refer to Table 2 for internal std., RPD criteria	• Review Internal Standards, as above • If RPD exceeds, reinject extract • Narrate any outliers.
		Matrix spike	As per client request	Refer to Tables 2 and 3	• Review data for usability • Narrate outliers
		Matrix spike duplicate	As per client request	Refer to Tables 2 and 3	• Review data for usability • Narrate outliers

TABLE B-8

CORRECTIVE ACTION SUMMARY FOR ICP METALS AND COLD VAPOR ATOMIC ABSORPTION ANALYSIS

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
METALS					
SW-846 6010A Inductively Coupled Plasma (ICP) and SW-846 7470/7471 Cold Vapor Atomic Absorption (CVAA)		Initial laboratory mixed standard multipoint calibration	Daily prior to analysis	$r \geq 0.995$	Recalibrate
		Calibration blank	After initial calibration and each subsequent calibration	< PQL	Clean system Rerun blank
		ICP interference check	Beginning of daily run, after 8 hours, and/or end of run sequence	80-120% of true value for EPA check sample elements	Verify calibration Verify IEC and update as necessary Recalibrate
		Initial calibration verification (ICV)	After calibration, prior to sample analysis	± 10% expected concentration	Reanalyze ICV Recalibrate
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 10% expected concentration	Reanalyze CCV Recalibrate Reanalyze affected samples back to the last acceptable CCV
		Method blank	1 per preparation batch (≤20 samples)	< PQL	Reanalyze Assess impact on data Redigest/reanalyze samples if blank fails twice
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	Reanalyze LCS Assess impact on data Redigest/reanalyze affected samples Narrate all outliers

TABLE B-9

WET CHEMISTRY ANALYSES
CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 7196 Colorimetric	Hexavalent Chromium	Initial multipoint calibration	Initially and as required	Correlation coefficient (r) ≥ 0.995	Recalibrate
EPA 351.2	Total Kjeldahl Nitrogen (TKN)	Initial calibration verification (ICV)	Daily, prior to sample analysis	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary Reanalyze all affected samples
		Calibration blank	Daily prior to sample analysis, and after each ICV and CCV	< PQL	Reanalyze blank Reanalyze samples back to last clean blank
		Method blank	1 per preparation batch (≤20 samples)	< PQL	Assess impact on data Reanalyze blank Redigest/reanalyze affected samples
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC criteria	Reanalyze LCS Assess impact on data Reprep/reanalyze samples as necessary Narrate all outliers
EPA 325.2 EPA 365.1 EPA 365.2	Chloride O-phosphorous Total phosphorous	Initial multipoint calibration	Initially and as required	Correlation coefficient (r) ≥ 0.995	Check calculations Recalibrate
EPA 353.2	Nitrate Nitrite	Initial calibration verification (ICV)	Prior to sample analysis	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary
EPA 340.2	Fluoride	Continuing calibration verification (CCV)	Every 10 samples and end of run	± 15% expected concentration	Reanalyze CCV Recalibrate as necessary
EPA 350.3 EPA 375.4 EPA 150.1 EPA 376.1 EPA 305.1	Ammonia Sulfate pH Sulfide Acidity	Calibration blank	Daily and after each ICV and CCV	< RL	Reanalyze blank Clean system Reanalyze samples back to last clean blank
		Method Blank	1 per preparation batch (≤20 samples)	< RL	Reanalyze reference standard Reanalyze ICV Recalibrate as necessary
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers

TABLE B-9

WET CHEMISTRY ANALYSES
CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	Assess impact on data Reanalyze LCS Reprep/reanalyze samples as necessary Narrate all outliers
		Method blank	1 per preparation batch (≤20 samples)	<PQL	Assess impact on data Reanalyze blank Reprep and analyze as necessary
EPA 415.1	Total Organic Carbon	(1) Two point calibration (2) Acid standardization	(1) Daily (2) Daily	(1) Measured value within ±0.1 pH unit (2) Duplicate standardizations agree to +/- 0.2 ml	(1a) Recalibrate (1b) See Instrument Manual (2a) Recheck Na_2CO_3 standard prepare fresh standard solution if necessary (2b) Prepare fresh acid solutions
		Method blank	1 per preparation batch	≤RL	(1) Assess impact on data (2) Reanalyze blank if necessary (3) Reprep/reanalyze as necessary
		Laboratory control sample	1 per batch	Refer to Table C-9	(1) Assess impact on data (2) Reanalyze LCS (3) Reprep/reanalyze as necessary
		Matrix spike	5%	Refer to Table C-9	(1) Assess data (4x rule) (2) Reanalyze once, if necessary (3) Report as footnoted
		Matrix duplicate	5%	Refer to Table C-9	Same as matrix spike

TABLE C-1

PRACTICAL QUANTITATION LIMITS FOR MODIFIED METHOD TO-13

Parameter	Method	Analyte	Air (µg/sample)
Semivolatile Organic Compounds	Mod TO-13	**Base/Neutral Extractables**	
		Acenaphthene	10
		Acenaphthylene	10
		Anthracene	10
		Benzo (a) anthracene	10
		Benzo (b) fluoranthene	10
		Benzo (k) fluoranthene	10
		Benzo (g,h,i) perylene	10
		Benzo (a) pyrene	10
		Benzyl alcohol	10
		Bis (2-chloroethoxy) methane	10
		Bis(2-ethylhexyl)phthalate	10
		Bis(2-chloroethyl)ether	10
		Bis(2-chloroisopropyl)ether	10
		4-Bromophenyl phenyl ether	10
		Butyl benzyl phthalate	10
		4-Chloroaniline	10
		2-Chloronaphthalene	10
		4-Chlorophenyl phenyl ether	10
		Chrysene	10
		Dibenz (a,h) anthracene	10
		Dibenzofuran	10
		Di-n-butylphthalate	10
		1,2-Dichlorobenzene	10
		1,3-Dichlorobenzene	10
		1,4-Dichlorobenzene	10
		3,3'-Dichlorobenzidine	20
		Diethyl phthalate	10
		Dimethyl phthalate	10
		2,4-Dinitrotoluene	10
		2,6-Dinitrotoluene	10
		Di-n-octyl phthalate	10
		Fluoranthene	10
		Fluorene	10
		Hexachlorobenzene	10
		Hexachlorobutadiene	10
		Hexachlorocyclopentadiene	10
		Hexachloroethane	10
		Indeno(1,2,3-cd) pyrene	10
		Isophorone	10
		2-Methylnaphthalene	10
		Naphthalene	10
		2-Nitroaniline	10
		3-Nitroaniline	50

TABLE C-1

PRACTICAL QUANTITATION LIMITS FOR MODIFIED METHOD TO-13

Parameter	Method	Analyte	Air (µg/sample)
Semivolatile Organic Compounds (continued)	Mod TO-13	4-Nitroaniline	50
		Nitrobenzene	10
		N-Nitrosodiphenylamine	10
		N-Nitrosodipropylamine	10
		Phenanthrene	10
		Pyrene	10
		1,2,4-Trichlorobenzene	10
		Acid Extractables	
		4-Chloro-3-methylphenol	10
		2-Chlorophenol	10
		2,4-Dichlorophenol	10
		2,4-Dimethylphenol	10
		4,6-Dinitro-2-methylphenol	50
		2,4-Dinitrophenol	50
		2-Methylphenol	10
		4-Methylphenol	10
		2-Nitrophenol	10
		4-Nitrophenol	50
		Pentachlorophenol	50
		Phenol	10
		2,4,5-Trichlorophenol	50
		2,4,6-Trichlorophenol	10

µg microgram

TABLE C-2

PRACTICAL QUANTITATION LIMITS FOR METHOD TO-14

Parameter	Method	Analyte	PPBV
Volatile Organic Compounds	TO-14 (Air)	Dichlorodifluoromethane	2
		Chloromethane	4
		1,2-Dichloro-1,1,2,2-Tetrafluoroethane	2
		Vinyl Chloride	2
		Bromomethane	2
		Chloroethane	4
		Trichlorofluoromethane	2
		1,1-Dichloroethene	2
		Carbon Disulfide	10
		1,1,2-Trichloro-1,2,2-Trifluoroethane	2
		Acetone	10
		Methylene Chloride	2
		trans-1,2-Dichloroethene	2
		1,1-Dichloroethane	2
		Vinyl Acetate	10
		cis-1,2-Dichloroethene	2
		2-Butanone	10
		Chloroform	2
		1,1,1,-Trichloroethane	2
		Carbon Tetrachloride	2
		Benzene	2
		1,2-Dichloroethane	2
		Trichloroethene	2
		1,2-Dichloropropane	2
		Bromodichloromethane	2
		cis-1,3-Dichloropropene	2
		4-Methyl-2-pentanone	10
		Toluene	2
		trans-1,3-Dichloropropene	2
		1,1,2-Trichloroethane	2
		Tetrachloroethene	2
		2-Hexanone	30
		Dibromochloromethane	2
		1,2-Dibromoethane (EDB)	2
		Chlorobenzene	2
		Ethylbenzene	2
		Total Xylenes	2
		Styrene	2
		Bromoform	2
		1,1,2,2-Tetrachloroethane	2
		Benzyl Chloride	10
		4-Ethyl Toluene	2
		1,3,5-Trimethylbenzene	2
		1,2,4-Trimethylbenzene	2
		1,3-Dichlorobenzene	2
		1,4-Dichlorobenzene	2
		1,2-Dichlorobenzene	2
		1,2,4-Trichlorobenzene	20
		Hexachlorobutadiene	4

PPBV Parts by billion by volume

TABLE C-3

VOLATILE ORGANIC COMPOUNDS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (25 ml purge) MDL µg/l	Water (25 ml purge) PQL µg/l	Soil MDL µg/kg	Soil PQL µg/kg
Volatile Organic Compounds	SW8260A	Acetone	2.80	5	4.4	50
		Acrylonitrile	0.27	1	1.4	5
		Allyl chloride	0.20	1	2.3	5
		Benzene	0.04	1	1.9	5
		Bromobenzene	0.05	1	0.6	5
		Bromochloromethane	0.19	1	1.0	5
		Bromodichloromethane	0.12	1	0.9	5
		Bromoform	0.11	1	0.7	5
		Bromomethane	0.25	1	2.3	10
		2-Butanone	2.30	5	5.1	10
		n-Butylbenzene	0.07	1	0.8	5
		tert-Butylbenzene	0.12	1	0.9	5
		sec-Butylbenzene	0.10	1	0.9	5
		Carbon disulfide	0.15	1	1.1	5
		Carbon tetrachloride	0.12	1	1.2	5
		Chlorobenzene	0.04	1	1.2	5
		Chlorodibromomethane	0.12	1	0.6	5
		Chloroethane	0.24	1	6.4	10
		2-Chloroethylvinyl ether	0.21	1	2.6	10
		Chloroform	0.17	1	1.0	5
		Chloromethane	0.12	1	1.6	10
		2-Chlorotoluene	0.12	1	0.6	5
		4-Chlorotoluene	0.17	1	0.8	5
		1,2-Dibromo-3-chloropropane	0.35	1	2.1	5
		1,2-Dibromoethane	0.15	1	1.3	5
		Dibromomethane	0.15	1	1.0	5
		1,2-Dichlorobenzene	0.15	1	0.7	5
		1,3-Dichlorobenzene	0.12	1	0.6	5
		1,4-Dichlorobenzene	0.06	1	0.8	5
		Dichlorodifluoromethane	0.12	1	1.6	10
		1,1-Dichloroethane	0.12	1	1.0	5
		1,2-Dichloroethane	0.17	1	1.1	5
		1,1-Dichloroethene	0.06	1	1.6	5
		cis-1,2-Dichloroethene	0.12	1	1.0	5
		trans-1,2-Dichloroethene	0.12	1	1.2	5
		1,2-Dichloroethene (Total)	0.24	1	NA	NA
		1,3-Dichloropropane	0.15	1	1.7	5
		2,2-Dichloropropane	0.15	1	1.6	5
		1,1-Dichloropropene	0.12	1	1.1	5
		cis-1,3-Dichloropropene	0.15	1	0.8	5
		trans-1,3-Dichloropropene	0.12	1	1.4	5
		cis-1,4-Dichloro-2-butene	0.31	1	2.6	5
		trans-1,4-Dichloro-2-butene	0.63	1	2.0	5
		Ethyl benzene	0.04	1	1.0	5
		Ethyl methacrylate	0.24	1	1.9	5
		Hexachlorobutadiene	0.05	1	1.3	5
		2-Hexanone	2.59	5	1.7	10
		Iodomethane	0.13	1	0.6	5
		Isopropylbenzene	0.18	1	0.8	5
		p-Isopropyltoluene	0.11	1	0.9	5
		Methacrylonitrile	0.86	1	2.9	5
		Methylene chloride	0.43	1	6.6	10
		Methyl methacrylate	0.31	1	1.1	5
		4-Methyl-2-pentanone	3.06	5	1.9	10

TABLE C-3

VOLATILE ORGANIC COMPOUNDS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (25 ml purge) MDL µg/l	Water (25 ml purge) PQL µg/l	Soil MDL µg/kg	Soil PQL µg/kg
Volatile Organic Compounds (continued)	SW8260A	Methyl-tert-butyl ether	0.15	1	1.6	5
		Naphthalene	0.14	1	1.2	5
		Pentachloroethane	0.13	1	0.8	5
		n-Propylbenzene	0.08	1	0.9	5
		Styrene	0.17	1	1.2	5
		1,2,3-Trichlorobenzene	0.13	1	1.1	5
		1,2,4-Trichlorobenzene	0.05	1	0.4	5
		1,1,1,2-Tetrachloroethane	0.06	1	0.8	5
		1,1,2,2-Tetrachloroethane	0.11	1	1.5	5
		Tetrachloroethane	0.07	1	0.6	5
		Toluene	0.08	1	0.7	5
		1,1,1-Trichloroethane	0.12	1	0.8	5
		1,1,2-Trichloroethane	0.17	1	1.4	5
		Trichloroethene	0.12	1	1.5	5
		Trichlorofluoromethane	0.07	1	0.6	5
		1,2,3-Trichloropropane	0.40	1	1.4	5
		1,3,5-Trimethylbenzene	0.04	1	0.9	5
		Vinyl acetate	0.28	1	1.6	10
		Vinyl chloride	0.08	1	1.2	10
		o-Xylene	0.07	1	0.5	5
		m,p-Xylene	0.10	2	0.7	5
		Xylenes (Total)	0.16	5	1.1	5

NA Not available; MDL study not required by SW-846
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-5

ORGANOCHLORINE PESTICIDES AND PCBS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Organochlorine Pesticides and PCBs	SW8081	Aldrin	0.050	0.012	8.0	5.37
		alpha-BHC	0.050	0.024	8.0	0.67
		beta-BHC	0.050	0.016	8.0	0.19
		delta-BHC	0.050	0.012	8.0	0.34
		Lindane	0.050	0.022	8.0	0.69
		Chlordane(technical chlordane)	0.50	0.024	80	NA
		4,4'-DDD	0.10	0.051	16	0.69
		4,4'-DDE	0.10	0.026	16	0.21
		4,4'-DDT	0.10	0.049	16	1.01
		Dieldrin	0.10	0.046	16	1.31
		Endosulfan I	0.050	0.018	8.0	0.86
		Endosulfan II	0.10	0.031	16	2.72
		Endosulfan sulfate	0.10	0.025	16	0.22
		Endrin	0.10	0.047	16	1.31
		Endrin aldehyde	0.10	0.042	16	1.10
		Heptachlor	0.050	0.030	8.0	0.82
		Heptachlor epoxide	0.050	0.012	8.0	0.42
		Methoxychlor	0.50	0.22	80	7.30
		Toxaphene	5.0	1.3	160	44.4
		PCB-1016	0.50	0.31	80	2.12
		PCB-1221	0.50	0.18	80	41.6
		PCB-1232	0.50	0.18	80	5.88
		PCB-1242	0.50	0.07	80	5.55
		PCB-1254	1.0	0.12	160	4.37
		PCB-1260	1.0	0.90	160	4.84

NA Not available; MDL study not required by SW-846
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-6

CHLORINATED HERBICIDES AND PENTACHLOROPHENOL ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (µg/l)	Soil (µg/kg)
Chlorinated Herbicides	SW 8150	Pentachlorophenol	1.0	NA

NA Not analyzed
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-7

DIOXIN/FURAN REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water[(a)] (pg/l)	Soil[(b)] (pg/g)
Dioxins/Furans	SW-8290	**DIOXINS**		
		TCDDs (Total)	0.80	0.21
		2,3,7,8-TCDD	0.80	0.21
		PeCDDs (Total)	1.3	2.4
		1,2,3,7,8-PeCDD	1.3	0.32
		HxCDDs (Total)	2.2	0.33
		1,2,3,4,7,8-HxCDD	2.0	0.33
		1,2,3,6,7,8-HxCDD	2.0	0.31
		1,2,3,7,8,9-HxCDD	2.3	0.33
		HpCDDs (Total)	2.0	0.28
		1,2,3,4,6,7,8-HpCDD	2.0	0.28
		OCDD	9.2	2.0
		FURANS		
		TCDFs (Total)	1.3	0.14
		2,3,7,8-TCDF	1.3	0.14
		PeCDFs (Total)	1.1	0.33
		1,2,3,7,8-PeCDF	1.1	0.33
		2,3,4,7,8-PeCDF	0.94	0.28
		HxCDFs (Total	2.0	0.25
		1,2,3,4,7,8-HxCDF	0.72	0.17
		1,2,3,6,7,8-HxCDF	0.57	0.21
		1,2,3,7,8,9-HxCDF	0.89	0.20
		2,3,4,6,7,8-HxCDF	2.6	0.25
		HpCDFs (Total)	3.9	0.20
		1,2,3,4,6,7,8-HpCDF	2.4	0.15
		1,2,3,4,7,8,9-HpCDF	3.9	0.20
		OCDF	6.1	0.27

pg/l Picogram per liter
pg/g Picogram per gram

(a) Based upon 1 liter sample aliquot. Sensitivity of the method depends on the level of interferences rather than instrument limitations. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

(b) Based upon 10 gram sample aliquot. Maximum CRQLs for samples "as received." Correction for moisture content may raise reporting limits above these levels. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

TABLE C-8

REPORTING LIMITS FOR TRACE METALS

Parameter	Analytical Method	Analyte	Water PQL (mg/l)	Water MDL (µg/l)	Soil PQL (mg/kg)	Soil MDL (mg/kg)
METALS	6010A	Antimony	.060	1.197	6.0	0.0006
	6010A	Arsenic	.010	1.335	1.0	0.0007
	6010A	Barium	0.200	.093	20	0.00005
	6010A	Beryllium	.005	0.112	0.5	0.0001
	6010A	Cadmium	.005	.171	0.5	0.0001
	6010A	Calcium	.500	161	500	16.2
	6010A	Chromium	.010	1.703	1	0.0002
	6010A	Iron	.100	9.71	10	0.0047
	6010A	Lead (Trace)	.003	1.064	0.3	0.001
	6010A	Magnesium	.500	3.71	500	0.0018
	6010A	Manganese	.015	0.238	1.5	0.0001
	7470/7471	Mercury	.002	0.06	0.1	0.00003
	6010A	Potassium	.500	17.2	500	0.0019
	6010A	Thallium	.010	2.074	1	0.001
	6010A	Selenium	.005	2.768	0.5	0.0007
	6010A	Silver	.010	.188	1	0.0002
	6010A	Sodium	5.0	561	500	0.28

mg/l Milligram per liter
µg/l Microgram per liter
mg/kg Milligram per kilogram

TABLE C-9

WET CHEMISTRY METHODS
REPORTING LIMITS

Analysis	Analytical[a] Method	Analyte	Water (mg/L)	Soil (mg/kg)
Common Anions	EPA 325.2	Chloride	0.4	10
	EPA 365.2	O-phosphorous	0.02	.2
	EPA 375.4	Sulfate	2.0	20
	EPA 365.2	T-phosphorous	0.02	0.2
	EPA 376.1	Sulfide	1.0	NA
Nitrate/Nitrite	EPA 353.2	Nitrate	0.02	0.2
		Nitrite	0.02	0.2
Fluoride	EPA 340.2	Fluoride	0.1	1.0
Total Organic Carbon	EPA 415.1	TOC	1.0	100
Ammonia	EPA 350.3	Ammonia	0.1	10
Hexavalent chromium	SW846-7196	Hexavalent Chromium	0.010	2.0
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	Total Kjeldahl Nitrogen (TKN)	0.1	100
Acidity	EPA 310.1	Acidity	10	10
pH	EPA 150.1/ SW846-9045	pH	0.1 (unit)	0.1 (unit)

mg/l Milligram per liter
mg/kg Milligram per kilogram
NA Not analyzed for this project

(a) The methods cited are from the following sources:

Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983—with additions)

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (USEPA, 1986)

TABLE D-1

SEMI-VOLATILE ORGANIC COMPOUND ANALYSIS FOR AIR QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

		Laboratory-Established Control Limits		
Analytical Method	**Spiking Compounds**	**Spike Concentration (µg/sample) Air**	**Accuracy Recovery (%) Air**	**Precision (RPD %) Air**
Laboratory Control Samples	Matrix			
VOC TO-13	Phenol	100	60-140	35
	2-Chlorophenol	100	60-140	35
	1,4-Dichlorobenzene	50	60-140	35
	N-Nitroso-di-n-propylamine	50	60-140	35
	1,2,4-Trichlorobenzene	50	60-140	35
	4-Chloro-3-methylphenol	100	60-140	35
	Acenaphthene	50	60-140	35
	4-Nitrophenol	100	60-140	35
	2,4-Dinitrotoluene	50	60-140	35
	Pentachlorophenol	100	60-140	35
	Pyrene	50	60-140	35
	Surrogate			
	Nitrobenzene-d5	50	46-121	NA
	2-Fluorobiphenyl	50	64-123	NA
	Terphenyl-d14	50	33-170	NA
	Phenol-d5	50	29-115	NA
	2-Fluorophenol	50	50-117	NA
	2,4,6-Tribromophenol	50	29-143	NA

NA Not applicable

TABLE D-2

VOLATILE ORGANIC COMPOUND ANALYSIS FOR AIR QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits: *Spike Concentration (ppbv) Air	Laboratory-Established Control Limits: Accuracy Recovery (%) Air	Laboratory-Established Control Limits: Precision (RPD %) Air
Laboratory Control Samples				
	Matrix			
VOC TO-14	Methylene chloride	50	86-116	10
	1,1-Dichloroethene	50	90-115	10
	Trichloroethene	50	85-114	10
	Toluene	50	92-114	10
	1,1,2,2-Tetrachloroethane	50	76-124	10

TABLE D-3

VOLATILE ORGANIC COMPOUND ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (µg/l)	Spike Concentration Soil/Sediments (µg/kg)	Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Matrix Spike/Matrix Spike Duplicate							
VOCs SW 8260A	<u>Matrix</u>						
	1,1-Dichloroethene	5.0	50	60-140	59-172	20	22
	Trichloroethene	5.0	50	60-140	62-137	20	24
	Benzene	5.0	50	60-140	66-142	20	21
	Toluene	5.0	50	60-140	59-139	20	21
	Chlorobenzene	5.0	50	60-140	60-133	20	21
	<u>Surrogate Spikes</u>						
	Toluene-d8	5.0	50	90-108	90-123	NA	NA
	Bromofluorobenzene	5.0	50	78-115	75-129	NA	NA
	1,2-Dichloroethane-d4	5.0	50	62-121	70-139	NA	NA
Laboratory Control Samples							
VOCs SW 8260A	<u>Matrix</u>						
	Bromomethane	5.0	20	10-200	10-200	NA	NA
	Vinyl chloride	5.0	20	10-200	10-200	NA	NA
	1,1-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	trans-1,2-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	cis-1,2-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	1,1-Dichloroethane	5.0	20	10-200	10-200	NA	NA
	Chloroform	5.0	20	10-200	10-200	NA	NA
	1,2-Dichloroethane	5.0	20	10-200	10-200	NA	NA
	1,1,1-Trichloroethane	5.0	20	10-200	10-200	NA	NA
	Carbon tetrachloride	5.0	20	10-200	10-200	NA	NA
	Bromodichloromethane	5.0	20	10-200	10-200	NA	NA

TABLE D-3

VOLATILE ORGANIC COMPOUND ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits: Spike Concentration Water (µg/l)	Spike Concentration Soil/Sediments (µg/kg)	Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
	1,2-Dichloropane	5.0	20	10-200	10-200	NA	NA
	Trichloroethene	5.0	20	10-200	10-200	NA	NA
	1,1,2-Trichloroethane	5.0	20	10-200	10-200	NA	NA
	Benzene	5.0	20	10-200	10-200	NA	NA
	Tetrachloroethene	5.0	20	10-200	10-200	NA	NA
	Toluene	5.0	20	10-200	10-200	NA	NA
	Chlorobenzene	5.0	20	10-200	10-200	NA	NA
	Ethylbenzene	5.0	20	10-200	10-200	NA	NA
	m-and p-Xylenes	5.0	20	10-200	10-200	NA	NA
	o-Xylene	5.0	20	10-200	10-200	NA	NA
	1,3-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	1,4-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	1,2-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	Surrogate Spikes						
	Toluene-d8	5.0	50	90-108	90-123	NA	NA
	Bromofluorobenzene	5.0	50	78-115	75-129	NA	NA
	1,2-Dichloroethane-d4	5.0	50	62-121	70-139	NA	NA

NA Not available

TABLE D-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (µg/l)	Spike Concentration Soil/Sediments (µg/kg)	Laboratory-Established Control Limits: Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Matrix Spike/Matrix Spike Duplicates							
BNAEs, SW 8270	Matrix						
	Phenol	50	1,667	12-110	26-90	42	35
	2-Chlorophenol	50	1,667	27-123	25-102	40	50
	1,4-Dichlorobenzene	50	1,667	36-97	28-104	28	27
	n-Nitroso-di-n-propylamine	50	1,667	41-116	41-126	38	38
	1,2,4-Trichlorobenzene	50	1,667	39-98	38-107	28	23
	4-Chloro-3-methylphenol	50	1,667	23-97	26-103	42	33
	Acenaphthene	50	1,667	46-118	31-137	31	19
	4-Nitrophenol	50	1,667	10-80	11-114	50	50
	2,4-Dinitrotoluene	50	1,667	24-96	28-89	38	47
	Pentachlorophenol	50	1,667	9-103	17-109	50	47
	Pyrene	50	1,667	26-127	35-142	31	36
	Surrogate Spikes						
	Nitrobenzene-d5	50	1,667	35-114	23-120	NA	NA
	2-Fluorobiphenyl	50	1,667	43-116	30-115	NA	NA
	Terphenyl-d14	50	1,667	33-141	18-137	NA	NA
	Phenol-d5	75	2,500	10-94	24-113	NA	NA
	2-Fluorophenol	75	2,500	21-100	25-121	NA	NA
	2,4,6-Tribromophenol	75	2,500	10-123	19-122	NA	NA
Laboratory Control Samples							
BNAE SW 8270	Phenol	50	1,700	30-112	10-112	NA	NA
	2-Chlorophenol	50	1,700	40-134	23-134	NA	NA
	1,4-Dichlorobenzene	50	1,700	40-124	61-90	NA	NA
	n-Nitroso-di-n-prop	50	1,700	40-138	52-93	NA	NA
	1,2,4-Trichlorobenzene	50	1,700	44-142	67-94	NA	NA
	4-Chloro-3-methylphenol	50	1,700	40-147	40-145	NA	NA
	Acenaphthene	50	1,700	47-145	63-92	NA	NA
	4-Nitrophenol	50	1,700	40-132	37-132	NA	NA

TABLE D-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (µg/l)	Spike Concentration Soil/Sediments (µg/kg)	Laboratory-Established Control Limits: Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Laboratory Control Samples (continued)							
BNAE SW 8270							
	2,4-Dinitrotoluene	50	1,700	40-139	67-95	NA	NA
	Pentachlorophenol	50	1,700	40-150	29-152	NA	NA
	Pyrene	50	1,700	52-115	48-121	NA	NA
	2,4-Dinitrophenol	50	1,700	10-141	10-133	NA	NA
	2,4-Dichlorophenol	50	1,700	39-135	10-167	NA	NA
	2,4-Dimethyphenol	50	1,700	32-119	10-167	NA	NA
	2,4,6-Trichlorophenol	50	1,700	37-144	10-159	NA	NA
	Napthalene	50	1,700	21-133	10-145	NA	NA
	2-Methylnaphthalene	50	1,700	10-136	54-93	NA	NA
	<u>Surrogate Spikes</u>						
	Nitrobenzene-d5	50	1,700	35-114	23-120	NA	NA
	2-Fluorobiphenyl	50	1,700	43-116	30-115	NA	NA
	Terphenyl-d14	50	1,700	33-141	18-137	NA	NA
	Phenol-d5	75	2,500	10-94	24-113	NA	NA
	2-Fluorophenol	75	2,500	21-100	25-121	NA	NA
	2,4,6-Tribromophenol	75	2,500	10-123	19-122	NA	NA

NA Not applicable

TABLE D-5

PESTICIDE/PCB ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits: Spike Concentration Water (µg/l)	Spike Concentration Soil/Sediments (µg/kg)	Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Matrix Spike/Matrix Spike Duplicates							
Pesticides/PCBs, SW 8081	Matrix						
	Lindane (Gamma -BHC)	0.20	16.0	32-127	32-127	15	50
	Heptachlor	0.20	16.0	34-111	34-111	20	31
	Aldrin	0.20	16.0	42-122	42-122	22	43
	Dieldrin	0.20	16.0	36-146	36-146	18	38
	Endrin	1.0	80.0	30-147	30-147	21	45
	4,4'-DDT	1.0	80.0	25-160	25-160	27	50
	Surrogate Spikes						
	TCMX	0.20	0.667	60-150	60-150	NA	NA
	DCB	0.20	0.667	60-150	60-150	NA	NA
Laboratory Control Samples							
Pesticides/PCB SW 8081	Matrix						
	Lindane (Gamma-BHC)	0.20	13	60-127	52-108	NA	NA
	Heptachlor	0.20	13	61-111	52-111	NA	NA
	Aldrin	0.20	13	64-113	42-122	NA	NA
	Dieldrin	0.20	13	75-146	57-146	NA	NA
	Endrin	1.0	13	58-133	55-126	NA	NA
	4,4-DDT	1.0	13	46-134	25-160	NA	NA
	Surrogate Spikes						
	TCMX	0.20	0.667	60-150	60-150	NA	NA
	DCB	0.20	0.667	60-150	60-150	NA	NA

NA Not applicable

TABLE D-6

SW-846 8150 ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits: Spike Concentration (µg/l)	Accuracy (% Recovery)	Precision (RPD %)
Matrix Spike/Matrix Spike Duplicates				
	Pentachlorophenol	5	30-140	35
	Surrogate 2,4-DB	5	30-140	NA
Laboratory Control Samples				
	Pentachlorophenol	5	30-140	NA
	Surrogate 2,4-DB	5	30-140	NA

TABLE D-7

DIOXIN/FURAN QUALITY CONTROL CRITERIA
FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil/Sediments (mg/kg)	Laboratory-Established Control Limits: Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Laboratory Control Samples							
SW-8290 (c)	2,3,7,8-TCDD	200	200	60-140	60-140	50	50
	2,3,7,8-TCDF	200	200	60-140	60-140	50	50
	1,2,3,7,8-PeCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8-PeCDF	500	500	60-140	60-140	50	50
	2,3,6,7,8-PeCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDF	500	500	60-140	60-140	50	50
	2,3,4,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDD	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDF	500	500	60-140	60-140	50	50
	1,2,3,4,7,8,9-HpCDF	500	500	60-140	60-140	50	50
	OCDD	1000	1000	60-140	60-140	50	50
	OCDF	1000	1000	60-140	60-140	50	50
(d)(f)	^{13}C-2,3,7,8-TCDD	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-2,3,7,8-TCDF	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,7,8-PeCDD	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,7,8-PeCDF	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,6,7,8-HxCDD	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HxCDF	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HpCDD	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HpCDF	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-OCDD	5000	5000	25-150	25-150	50	50

(c) Native compound limits are COE default limits.
(d) Method default control limits. Signal-to-noise is also evaluated for data acceptability.
(f) These labeled analytes are spiked into all samples. RPD will be used to compare sample/duplicate IS recoveries.

TABLE D-7

DIOXIN/FURAN QUALITY CONTROL CRITERIA
FOR LABORATORY DATA EVALUATION
(CONTINUED)

Analytical Method	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil/Sediments (mg/kg)	Laboratory-Established Control Limits: Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Laboratory Control Samples (continued)							
SW-8290 (c)	2,3,7,8-TCDD	200	200	60-140	60-140	50	50
	2,3,7,8-TCDF	200	200	60-140	60-140	50	50
	1,2,3,7,8-PeCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8-PeCDF	500	500	60-140	60-140	50	50
	2,3,6,7,8-PeCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDF	500	500	60-140	60-140	50	50
	2,3,4,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDD	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDF	500	500	60-140	60-140	50	50
	1,2,3,4,7,8,9-HpCDF	500	500	60-140	60-140	50	50
	OCDD	1000	1000	60-140	60-140	50	50
	OCDF	1000	1000	60-140	60-140	50	50
(d)	^{13}C-2,3,7,8-TCDD	1000	1000	25-150	25-150	50	50
(d)	^{13}C-2,3,7,8-TCDF	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,7,8-PeCDD	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,7,8-PeCDF	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,6,7,8-HxCDD	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HxCDF	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HpCDD	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HpCDF	2500	2500	25-150	25-150	50	50
(d)	^{13}C-OCDD	5000	5000	25-150	25-150	50	50

(c) Native compound limits are COE default limits.
(d) Method default limits. Signal-to-noise is also evaluated for data acceptability.
(f) These labeled analytes are spiked into all samples. RPD will be used to compare sample/duplicate IS recoveries.

TABLE D-8

ICP AND COLD VAPOR ATOMIC ABSORPTION METALS ANALYSES QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (µg/L)	Spike Concentration Soil/Sediments (mg/kg)	Control Limits: Accuracy Percent Recovery (%) Water	Control Limits: Accuracy Percent Recovery (%) Soil/ Sediments	Control Limits: Precision (RPD %) Water	Control Limits: Precision (RPD %) Soil/ Sediments
Matrix Spike/Matrix Duplicates							
Metals ICP, 6010A	Antimony	500	50	80-120	95 ConfW	20	20
	Arsenic	2,000	200	80-120	95 ConfW	20	20
	Barium	2,000	200	80-120	95 ConfW	20	20
	Beryllium	50	5	80-120	95 ConfW	20	20
	Cadmium	50	5	80-120	95 ConfW	20	20
	Chromium	200	20	80-120	95 ConfW	20	20
	Lead (Trace)	500	50	80-120	95 ConfW	20	20
	Manganese	500	50	80-120	95 ConfW	20	20
	Selenium	2,000	200	80-120	95 ConfW	20	20
	Silver	50	5	80-120	95 ConfW	20	20
	Thallium	2,000	200	80-120	95 ConfW	20	20
	Calcium	2,000	10	80-120	95 ConfW	20	20
	Iron	2,000	10	80-120	95 ConfW	20	20
	Magnesium	2,000	10	80-120	95 ConfW	20	20
	Potassium	5,000	10	80-120	95 ConfW	20	20
	Sodium	10,000	10	80-120	95 ConfW	20	20
Metals, 7470/7471	Mercury	1.0	0.5	80-120	95 ConfW	20	20
Laboratory Control Samples							
Metals ICP, 6010A	Antimony	500	100	80-120	95 ConfW	20	20
	Arsenic	2,000	400	80-120	95 ConfW	20	20
	Barium	2,000	400	80-120	95 ConfW	20	20
	Beryllium	50	10	80-120	95 ConfW	20	20
	Cadmium	50	10	80-120	95 ConfW	20	20
	Chromium	200	40	80-120	95 ConfW	20	20
	Lead (Trace)	500	100	80-120	95 ConfW	20	20
	Manganese	500	100	80-120	95 ConfW	20	20
	Selenium	2,000	400	80-120	95 ConfW	20	20

TABLE D-8

ICP METALS ANALYSES
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION
(CONTINUED)

				Control Limits			
		Spike Concentration		Accuracy Percent Recovery (%)		Precision (RPD %)	
Analytical Method	Spiking Compounds	Water (µg/L)	Soil/Sediments (mg/kg)	Water	Soil/ Sediments	Water	Soil/ Sediments
Laboratory Control Samples (continued)							
Metals ICP, 6010A (continued)	Silver	50	10	80-120	95 ConfW	20	20
	Thallium	2,000	400	80-120	95 ConfW	20	20
	Calcium	5,000	1,300	80-120	95 ConfW	20	20
	Iron	5,000	9,000	80-120	95 ConfW	20	20
	Magnesium	5,000	1,100	80-120	95 ConfW	20	20
	Potassium	5,000	1,500	80-120	95 ConfW	20	20
	Sodium	5,000	300	80-120	95 ConfW	20	20
Metals, 7470/7471	Mercury	1.0	2.0	80-120	95 ConfW	20	20

95 ConfW 95% confidence window

TABLE D-9

WET CHEMISTRY ANALYSES
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil/Sediments (mg/kg)	Laboratory-Established Control Limits: Accuracy Percent Recovery (%) Water	Accuracy Percent Recovery (%) Soil/Sediments	Precision (RPD %) Water	Precision (RPD %) Soil/Sediments
Matrix Spike/Matrix Duplicates							
TOC, EPA 415.1	Potassium hydrogen phosphorous	.20	40	80-120	80-120	20	20
Hexavalent chromnium, 7196	Hexavalent chromium	25	25	80-120	80-120	20	20
Common Anions							
EPA 325.2	Chloride	25	25	80-120	80-120	20	20
EPA 365.2	O-phosphate	0.25	25	75-125	75-125	20	20
EPA 365.2	Total phosphorous	0.25	25	75-125	75-125	20	20
EPA 375.4	Sulfate	10	100	75-125	75-125	20	20
EPA 353.2	Nitrate	0.02	0.2	75-125	75-125	20	10
	Nitrite	0.02	0.2	75-125	75-125	20	20
EPA 340.2	Fluoride	2.0	20	75-125	75-125	20	20
EPA 350.3	Ammonia	2.0	2.0	75-125	75-125	20	20
EPA 351.2	TKN	NA	7	NA	75-125	NA	20
EPA 376.1	Sulfide	NA	NA	NA	NA	NA	NA
EPA 150.1	pH	NA	NA	NA	NA	NA	NA
EPA 305.1	Acidity	20	200	NA	80-120	20	20
Laboratory Control Samples							
TOC, EPA 415.1	Potassium hydrogen phosphorous	10	NA	80-120	NA	20	NA
Hexavalent chromnium, 7196	Hexavalent chromium	.25	NA	80-120	NA	20	NA
Common Anions							
EPA 325.2	Chloride	50	NA	80-120	NA	20	NA
EPA 365.2	O-phosphate	0.4	NA	80-120	NA	20	NA
EPA 365.2	Total phosphorous	0.4	NA	80-120	NA	20	NA
EPA 375.4	Sulfate	NA	NA	NA	NA	NA	NA
EPA 353.2	Nitrate	0.8	NA	80-120	NA	20	NA
	Nitrite	0.8	NA	80-120	NA	20	NA
EPA 340.2	Fluoride	4.0	NA	80-120	NA	20	NA
EPA 350.3	Ammonia	2.0	NA	80-120	NA	20	NA
EPA 351.2	TKN	NA	NA	NA	NA	NA	NA
EPA 376.1	Sulfide (duplicate sample)	NA	NA	80-120	NA	20	NA
EPA 305.1	Acidity	NA	NA	NA	NA	NA	NA
EPA 150.1	pH	NA	NA	NA	NA	NA	NA

NA Not applicable